A STUDY OF COMPOSITE ACTION
IN MATERIALS AFTER TREATMENT
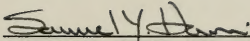
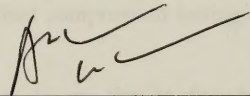Sarah Elizabeth Gray

A THESIS

in

Historic Preservation

Presented to the Faculties of the University of Pennsylvania in
Partial Fulfillment of the Requirements for the Degree of
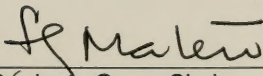
MASTER OF SCIENCE

2000

_____
Supervisor
Samuel Y. Harris, PE, AIA, Esq.
Adjunct Professor in Architecture

_____
Reader
Dr. A. Elena Charola
Lecturer in Historic Preservation

_____
Graduate Group Chair
Frank G. Matero
Associate Professor of Architecture

ACKNOWLEDGEMENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

ii

iv

LIST OF FIGURES

LIST OF TABLES

ABSTRACT

It has only been recently that researchers in conservation have investigated composite and interface action in treated porous building materials. This thesis aims to add to the body of knowledge examining the composite properties and mechanical performance of treated substrates. Discussion of deterioration mechanisms of porous building materials illustrates why conservation treatments are often necessary. The properties of chemical consolidants are discussed, with particular attention given to physical models of consolidation. Effects of deterioration and consolidation are illustrated graphically with strength profiles. After reviewing basic principles of material mechanics and structural analysis, a case study analyzes mechanical strengthening and composite action of load-bearing consolidated substrates. Although surface consolidation is not usually meant to structurally strengthen a substrate, researchers have shown that even partial penetration of consolidant does increase the substrate's mechanical properties. As part of an original testing program, 2" (5 cm) concrete cubes and 6" (15.2 cm) diameter by 12" (30.5 cm) concrete cylinders were produced. Specimen were left untreated, were fully-impregnated (in the case of the cubes), or were partially-impregnated (in the case of the cylinders) with a silicate ester consolidant. The specimens were subjected to compression testing in order to determine their compressive strengths and modulii of elasticity. The testing showed that full- and partial-depth consolidation increases the compressive strength of concrete. The modulus of elasticity of the treated cubes was higher than that of the untreated cubes. The modulus of elasticity of the treated cylinders was not significantly higher than that of the untreated cylinders. Shear and debonding failure was seen in some of the specimen, but this type of failure's link to consolidation was inconclusive. The testing program showed that composite and interface action may not be of great concern in undeteriorated treated specimen. Mathematical equations, however, show that if deterioration occurs or continues behind a shallow treatment zone, load-bearing structural elements may fail in compression when thin shells of consolidated material must carry the entire load applied to the structural element. Quantitative analysis of treated materials done in this thesis illustrates and emphasizes the need for complete penetration of deteriorated zones of material so that spalling of the treated shell or failure of the entire structure can be avoided.

CHAPTER ONE: INTRODUCTION

Collaboration in Historic Preservation

The field of historic preservation is interdisciplinary, bringing together architects, engineers, conservators, historians, construction tradespersons, chemists, geologists, real estate developers, and, at times, politicians. Such collaboration integrates all aspects of a restoration or rehabilitation program essential for the successful use of an historic building.

An architect generally directs the preservation of the building as a whole, while a conservator is charged with preserving surfaces or elements of the building. Before the architect and conservator begin to consider their preservation strategies for the building, an engineer often must assess the building's structural integrity. A structural engineer examines the condition of the building's materials to establish their current state, and analyzes the existing structural system to ascertain its adequacy for imposed loads. An engineer must also assess any structural alterations proposed for the building.

A preservation project cannot be approached such that when the work of the engineer is finished, that of the architect begins; or that when the work of the architect is finished, that of the conservator begins. Communication between project team members must continue during the entire preservation process: material treatments suggested by the conservator should be discussed with both the architect and the engineer, since these treatments could affect both the aesthetic and structural qualities of the substrate.

A building's historic associations may inspire its preservation, but current deterioration mechanisms and future material interventions will dictate the success or failure of its ultimate physical preservation. One of the desired goals of a preservation project is to maintain the building's historic visual character. In some instances, historic material must be strengthened, supported, or replaced in order for it to be safe for occupancy or to mitigate further material deterioration. During such intervention work, introducing new materials must be limited as much as possible in order to retain authenticity of the existing material and structure. Above all, the intervention measures should not be deleterious to the building's immediate or future preservation. Technical understanding and rigorous review of proposed uses or interventions ensures the preservation of the building. Interdisciplinary collaboration and review facilitate design and implementation of necessary stabilization work that is within the preservation philosophy prescribed for the building.

Objective of This Thesis

Giorgio Torraca stated, "Interdisciplinary' is frequently used as a word but is seldom put into real practice. Nevertheless, effective interdisciplinary work is an absolute requirement for progress in conservation."[1] In a Dahlem Workshop report, J.M. Teutonico advertised the need for specialized training for engineers wishing to work in

---

[1] Giorgio Torraca, "The Scientist's Role in Historic Preservation with Particular Reference to Stone Conservation," *Conservation of Historic Stone Buildings and Monuments* (Washington, DC: National Academy Press, 1982), 16.

2

the field of conservation.[2]   In order to promote interdisciplinary work between the engineering and conservation fields, this thesis offers an engineering perspective, review, and analysis of one type of conservation treatment and how that treatment may or may not affect the structural performance of its substrate. This thesis, then, bridges between work done by conservators and by engineers.   It supports progress not only in the conservation field, as Torraca suggested, but also in the engineering field.

This thesis focuses on one specific practice in the preservation field: the application of consolidating treatments to deteriorated porous building materials. Particular attention will be given to the deterioration and treatment of stone masonry, and a testing program using concrete as a generic substrate will be carried out. Much attention and research has already been dedicated to the characterization and testing of consolidating treatments prior to their application on deteriorated porous materials. Past work has examined changes in the chemical, physical, and mechanical properties of deteriorated masonry after treatment. Wendler reports that investigation of masonry conservation products indicates that there may be disadvantages and even risks that have not been considered during the many years of these treatments' application.[3]

---

[2] J.M. Teutonico, et al. "Group Report: How Can We Ensure the Responsible and Effective Use of Treatments (Cleaning, Consolidation, Protection)?" in *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage (West Sussex, England: John Wiley & Sons Ltd, 1997), 309.

[3] E. Wendler, "Materials and Approaches for the Conservation of Stone," in *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures*, edited by N.S. Baer and R. Snethlage (West Sussex, England: John Wiley & Sons Ltd, 1997), 181.

It has only been recently, however, that conservation researchers have investigated the effects of composite and interface mechanical properties between treated and untreated zones of porous building materials. This thesis aims to add to the body of knowledge examining the composite properties and mechanical performance of treated substrates. Furthermore, this thesis includes quantitative structural analysis of a treated load-bearing element. Such quantitative analysis will help explain and clarify what is usually explained qualitatively in the literature.

Before examining the effects of a consolidating treatment on porous building materials, this report will first discuss why and how these materials deteriorate, and why it is deemed necessary to slow this deterioration with a conservation treatment. Next, the purposes and types of conservation treatments – specifically consolidants – and how they mitigate deterioration will be reviewed briefly. In order to understand what any building material (undeteriorated, deteriorated, or treated) is required to do structurally, a review of engineering mechanics and structural analysis fundamentals will be provided. This introduction to deterioration, treatment, and structural mechanics will provide a background for understanding and analyzing the performance of treated, composite materials.

Consolidants: How Do They Affect Structural Performance?

As shown in the previous work of many researchers, differences in the mechanical properties of treated and untreated zones of building stone do in fact exist. The goal of treatment, after all, is to improve the deteriorated material. When a zone of treated

4

material exists in the element, it is different and presumably "better" than the untreated zone. Consolidation "improves" porous building material. Surface-applied consolidants usually reintegrate flaking and powdering grains of the material. Deep impregnation of consolidants, epoxies, or adhesives structurally strengthens its substrate. In some published studies, partial impregnation with a surface-applied consolidant has strengthened the substrate by increasing its compressive, tensile and/or bending strength.

This thesis examines the hypothesis that the mechanical properties of the treated and untreated zones of a consolidated substrate could be so different that detrimental forces in the material result. These forces, then, may affect the structural performance of the building element. A fictitious structural analysis case study included in this thesis illustrates the structural performance of a building element before and after consolidation. A laboratory testing program tests the hypothesis of this thesis and attempts to illustrate the behavior predicted in the fictitious case study. The testing examines the mechanical properties of an untreated material, a fully-impregnated treated material, and a composite material having both untreated and treated zones.

Such examination, testing, and analysis of composite action in materials after treatment is lacking in the current body of conservation treatment literature. This thesis should provide an interesting point of departure for further collaborative projects between engineering and conservation professionals in the historic preservation field.

CHAPTER TWO: DETERIORATION OF POROUS BUILDING MATERIALS

Introduction

Masonry built of a porous building material often lends a sense of durability and permanence to buildings constructed from it. Great masonry temples, cathedrals, and monuments somehow seem powerful or authoritative in appearance when reviewed by critics or observed by visitors. When porous building materials begin to deteriorate, this sense of permanence or "power" of the building may also deteriorate. The effect of deterioration may range from being aesthetically unpleasing to being deleterious to the structural integrity of the building.

Deterioration of porous building materials is an important contemporary issue, since many masonry structures are now exhibiting alarming deterioration and are undergoing conservation measures. Most conservation research focuses on the deterioration and treatment of natural stone. Lewin and Charola maintain that "the essential first stage of any stone conservation project is the identification of the nature of the stone decay, its rate of deterioration, and the causes of the decay."[1] From these assessments, an architect can propose architectural remedies of this deterioration, an engineer can analyze the effect of the deterioration on the performance of any load-bearing stone structural elements, and a conservator can suggest treatments to slow the deterioration process.

---

[1] S.Z. Lewin, and A.E. Charola, "Stone Decay Due to Foreign Inclusions," in *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi (Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981), 206.

For this thesis, research was directed mainly on the deterioration of natural stone, but much of what is described can be applied to all porous building materials.

Properties of Porous Building Materials

Understanding the properties of a porous building material is quite important in choosing its architectural use. These properties can also influence how and why the material weathers and deteriorates, and how that deterioration can be mitigated. The combination of material constituents, texture, porosity, and chemical composition influences the properties of the building material. In natural stones, the many ways of geologic formation leads to a complex variety of stones, each type having distinct properties and behaviors. Geology texts offer more in-depth descriptions of the formative processes and properties of stone, as well as deterioration processes of each stone type.[2] Properties of other porous building materials such as brick, concrete, and terra cotta are found in many available reference texts.

Physical Properties

Important physical properties of porous building materials include porosity, pore structure, pore size distribution, texture, and material composition. These properties can be quantified by laboratory tests, or estimated by comparison with published values for similar material.

---

[2] Alan Kehew's *Geology for Engineers & Environmental Scientists* and E.M. Winkler's *Stone: Properties, Durability in Man's Environment* are excellent sources for geology and stone deterioration information.

The pore characteristics of both deteriorated and undeteriorated material are important factors to consider when prescribing the type and application technique of a conservation treatment. Pore characteristics should also be studied after conservation treatment. Almost all published case studies in stone deterioration and conservation found in the bibliography of this thesis do include some form of pore characteristic quantification of the studied stone.

Chemical Properties

The chemical composition of a porous building material determines its resistance to several types of deterioration mechanisms. Silicate material in brick, terra cotta, and concrete is usually considered quite resistant to deterioration. In sedimentary stone, the chemical composition of the material cementing its constituent grains often dictates the susceptibility of the stone to deterioration. For example, carbonate stones like limestone are susceptible to chemical (acid) dissolution, while siliceous sandstones are usually more resistant to deterioration.

Mechanical Properties

Mechanical properties of the material define its ability to resistant loads. Such properties are useful in determining the appropriateness of using the material in a load-bearing structural element. Mechanical properties often result from both the physical structure and chemical composition (e.g., the strength of the chemical bonds and the chemical structure of material) of the material. Important mechanical properties include compressive strength, tensile strength, bending strength, modulus of rupture, and

modulus of elasticity. Table 2.1 offers approximate values for some physical, chemical and mechanical properties of natural stone.

Some Physical, Chemical and Mechanical Properties of Selected Stones

Stone	Class	Density	Porosity	Chemical Composition	Compressive Strength	Modulus of Elasticity
Granite	igneous	160–180 lb/ft^3 (2.56–2.88 g/cm^3)	0.5-1.5%	siliceous	14,000-45,000 psi (100-300 N/mm^2)	1.4x10^6 – 10x10^6 psi (10.000–70,000 N/mm^2)
Limestone	sedimentary	110–180 lb/ft^3 (1.76-2.90 g/cm^3)	0.3-3.0%	carbonaceous	2000-37,000 psi (1.4-260 N/mm^2)	1.4x10^6 – 10x10^6 psi (10,000–70,000 N/mm^2)
Sandstone	sedimentary	160-170 lb/ft^3 (2.56-2.70 g/cm^3)	0.5-3.0%	siliceous grains	4300-28,600 psi (30-200 N/mm^2)	0.6x10^6 – 10x10^6 psi (4000-40,000 N/mm^2)
Marble	metamorphic	150-200lb/ft^3 (2.37-3.20 g/cm^3)	0.6-2.3%	carbonaceous	3000-15,000 psi (21-100 N/mm^2)	0.3x10^6-12x10^6 psi (4200-84,000 N/mm^2)

Table 2.1

Quantitative data taken from Weber and Zinsmeister, *Conservation of Natural Stone: Guidelines to Consolidation, Restoration and Preservation* (Ehningen, Germany: expert verlag, 1991), 23-25; and E.M. Winkler, *Stone: Properties, Durability in Man's Environment* (New York: Springer-Verlag, 1975), 40-47.

Causes of Deterioration in Porous Building Materials

Deterioration is defined as those changes in physical, chemical or mechanical properties due to weathering that are seen as undesirable. Durability is defined as the degree of resistance to weathering and deterioration over time.[3]

Deterioration of porous building materials is determined by a combination of the material's durability, the extent and severity of its environment, and its weathering history before use in construction (so-called "inheritance").[4] The explicit causes of

---

[3] A.V. Turkington, "Stone Durability," in *Processes of Urban Decay, Proceedings of SWAPNET '95,* edited by Bernard J. Smith and Patricia A. Warke (London: Donhead Publishing, Ltd., 1996), 19-20.

[4] P.A. Warke, "Inheritance Effects in Building Stone Decay," in *Processes of Urban Decay, Proceedings of SWAPNET '95,* edited by Bernard J. Smith and Patricia A. Warke (London: Donhead Publishing, Ltd., 1996), 33.

deterioration are numerous and depend on the type and quality of the material under examination. Factors affecting deterioration can be classified as being intrinsic or extrinsic.[5] The interaction between intrinsic and extrinsic factors causes deterioration.

Intrinsic Factors of Deterioration

Intrinsic factors of deterioration are determined by the origin and nature of the material or by its use in a building. The material's physical, chemical, and mechanical properties of will affect the type and rate of deterioration. In particular, internal mineralogical weakness or incompatible foreign inclusions can influence a material's response to the environment. Materials of higher porosity and permeability presumably allow more deleterious agents to enter its volume. The chemical composition of the material may lend itself to dissolution upon exposure to acidic agents, or to oxidation upon atmospheric exposure. Furthermore, chemical alteration of the material due to environmental factors may lead to subsequent changes in its physical and mechanical properties. Related to its mechanical properties, use of an inherently weak material in a structural function may cause premature deterioration and failure of that material.

Extrinsic Factors of Deterioration

Extrinsic factors and causes of deterioration are determined by natural environmental agents having prolonged action, by natural agents occurring with sudden action, and by human activity.

---

[5] Guglielmo De Angelis D'Ossat, *Guide to the Methodical Study of Monuments and Causes of Their Decay* (Rome: ICCROM, 1982), 16-19.

Natural Agents with Prolonged Action

Water is considered to be one of the most deleterious natural agents causing deterioration. Water in the solid, liquid or gaseous state can be damaging when it lies on the material's surface or as it is carried into its pore structure. Water can carry agents (particularly acids and salts) that can lead to physical deterioration or to chemical dissolution. Finding the source of water entering the material is an important part of assessing and preventing deterioration.

Environmental exposure can be quite damaging to porous building materials. Wet-dry cycling, thermal cycling (freeze-thaw and diurnal heating), and salt cycling (hydration-dehydration) can all lead dimensional instability and pressures that can physically damage the material. Wind-born particulates can physically abrade exposed surfaces. Prolonged interference by biological growth can also physically abrade or erode exposed surfaces; in some cases, biological growth can chemically alter natural stone.

Natural Agents with Sudden Action

Earthquakes, floods, tornadoes, hurricanes and even fire can cause deterioration or even immediate failure of building elements. The occurrence of such events is often unpredictable, but by designing for their possible occurrence, damage to the building material can be prevented.

Human Activity

Environmental pollution caused by human activity (burning fossil fuels or releasing chemicals into the atmosphere or water supply) can cause or exacerbate naturally occurring deterioration processes. Winkler notes, "The urban atmosphere of the twentieth century creates special environmental problems to exposed stone and can accelerate the process of weathering to many times that of natural rural environments."[6]

Cutting and dressing of natural stone for construction or decorative purposes can induce stresses or lead to cracking of otherwise sound stone. Rusting and subsequent expansion of reinforcement or adjacent architectural features can stress all building materials and lead to their undue deterioration. Structural movement (foundation settlement and beam or column deflection) can lead to cracking. The use of inappropriate mortar or other detailing material can crack masonry or lead to deposition of damaging chemicals. Constructing or demolishing buildings adjacent to buildings can alter their micro-environment to a degree which can accelerate (or even decelerate) deterioration. Vibration or shock caused by mechanical equipment or even by bombs can induce deteriorating stresses in building material. Application of inappropriate cleaning, maintenance, or conservation techniques can also cause or accelerate deterioration.

---

[6] E.M. Winkler, *Stone: Properties, Durability in Man's Environment* (New York: Springer-Verlag, 1975), 88.

12

Mechanisms and Forms of Deterioration in Porous Building Materials

As suggested in the previous section, mechanisms of deterioration depend on intrinsic and/or extrinsic factors, and are outlined below.[7] These mechanisms can occur on a macroscopic or microscopic scale.

- *Wedging action* caused by foreign inclusions or deposits, incompatible constituents, or intrusive biological growths.
- *Volumetric change* of constituent materials or foreign inclusions (minerals or salts) due to water absorption, hydration, hygroscopic sorption, drying, oxidation, or temperature change.
- *Solution* (leaching) of constituents or binding cements into water.
- *Dissolution* of selected constituents, binding cements or of the entire material by chemicals introduced by water, the atmosphere or adjacent architectural details.
- *Chemical alteration* of constituents including oxidation, reduction, hydrolysis, hydration/dehydration of minerals or salts, or formation of secondary minerals.
- *Abrasion* by wind-born particles or human activity.
- *Deposition* of water- or air-born chemicals, or of conservation treatments that alter the material's chemistry or that lead to the formation of surface crusts.
- *Stress production* by freezing pore water, salt crystallization, differential expansion of constituents, or excessive structural loading.
- *Stress relief* after production, or in the case of stone, after exposure to the ground surface during weathering or quarrying.
- *Shock and vibration* caused by earthquakes, mechanical equipment, construction, and traffic.

The mechanisms of deterioration are manifested in one or more forms of deterioration. Deterioration can be observable on the material's exposed surfaces, observable after its interior cross-section is revealed, or observable only with microscopic examination.

---

[7] J. Rodrigues Delgado proposed some of these mechanisms in his article "Causes, Mechanisms and Measurement of Damage in Stone Monuments," 132. Other deterioration mechanisms were compiled from other sources or from the author's personal experience.

Again, the form of deterioration is often tied to the physical, chemical or mechanical properties of the material, as well as the intrinsic and extrinsic factors causing the deterioration. The following list defines several forms of deterioration; those listed are those usually remedied by conservation treatment.[8] Many of these deterioration forms apply only and specifically to natural stone.

- *Blistering* is the swelling and rupturing of a thin uniform skin. Caused by differential volumetric change or stress production.
- *Cracking* (micro and macro) is the rupturing of stone material. Caused by unaccommodated wedging action, volumetric change, stress production, stress relief, or shock.
- *Deformation* under load occurs before cracking. Caused by wedging action, volumetric change, stress production, stress relief, or shock.
- *Delamination* occurs when the outer surface splits apart into thin layers and peels off the exposed face. Caused by wedging action, volumetric change, chemical alteration, stress production, stress relief, or deposition of agents causing surface crusts.
- *Exfoliation* is the peeling, scaling or flaking of the surface in thin layers (similar to delamination).
- *Flaking* is the detachment of small, thin flat pieces of stone (is an early stage of exfoliation, delamination or spalling.)
- *Pitting* is a more localized form of sugaring or disintegration of stone, or loss of inclusions. Caused by volumetric change, stress production, stress relief, solution, dissolution, or abrasion.
- *Spalling* occurs when the outer layer or layers of stone break off unevenly or peel off in parallel layers. Caused by chemical alteration, stress production or stress relief.
- *Sugaring* (also granular disaggregation, sanding or powdering) is the gradual disintegration of the surface. Caused primarily by solution or dissolution whereby cementing material is lost, allowing the detachment of grains; also caused by loss of mechanical interlocking of constituent mineral grains or abrasion.
- *Surface crusts* (or scales) are formed by the deposition of chemicals that react with the material, or by the solution or dissolution of constituent minerals and subsequent precipitation of different and harder substances. Caused by solution, dissolution, chemical

---

[8] Anne E. Grimmer's *A Glossary of Historic Masonry Deterioration Problems and Preservation Treatments* (Washington, DC: Department of the Interior, National Parks Service, 1984) provides many of these terms and definitions.

14

deposition, and chemical alteration. Surface crusts can often hide other forms of deterioration behind them.

Assessing Material Deterioration

Evaluating a building material and quantifying its condition determine the type and extent of deterioration, providing a basis for subsequent structural analysis and conservation treatment. Evaluation of presumably deteriorated material should be compared to that of unweathered material of the same type and origin. Because color and texture are commonly affected by deterioration, visual inspection is the first and often simplest method of evaluating deterioration. Lab assessment and/or *in situ* field analysis can provide more detailed information about deterioration. The inspection, assessment, and analysis process does not and cannot follow any one "recipe," and each case or material type should have an inspection regime catered to it.

Both ASTM (American Society for Testing and Materials) and RILEM (Réunion Internationale des Laboratoires d'Essaies et de Recherches sur les Matériaux et les Constructions) specify many tests that aid in determining existence or extent of deterioration.[9] Such tests include visual examination, measurement of porosity and pore size distribution, water absorption capacity, and measurement of surface hardness, among many others. Determining the mechanical strength of deteriorated material is useful in illustrating the presence and effect of deterioration. Many of these tests are destructive;

---

[9] ASTM's tests are grouped by material type and are updated periodically. RILEM's Protection and Erosion of Monuments (PEM) Group defined physical methods of studying deterioration in 1978.

15

that is, samples of the material must be taken for evaluation. Non-destructive tests (NDTs) are of increasing popularity, and can include photogrammetry, X-ray tomography, ground penetrating radar, nuclear magnetic resonance pulse velocity or resonance frequency tests (to determine homogeneity of the material), and infrared thermography.[10] NDTs can often provide information about the material beneath its surface, where visual observation does not show any forms of external deterioration.

Interestingly, measuring the strength of a building material does not indicate its potential resistance to weathering or deterioration.[11] Instead, salt crystallization, freeze-thaw cycling, and wet-dry cycling provide information about the resistance of a sound, deteriorated, or treated material to weathering and deterioration. Although often informative, these tests are not without inherent problems. A large body of literature about these tests exists and should be consulted prior to their use in a research program.

An interesting phenomenon found in natural stone is that several types of stone are anisotropic; that is, their properties depend on the sample's orientation. This fact complicates not only testing of stone for material and deterioration properties, but it also makes understanding the mechanism of deterioration more complex. Zezza's article

---

[10] C.A. Price discusses available techniques in *Stone Conservation: An Overview of Current Research.* Medgyesi, Bleuer, and Kraisell offer a methodology and application of some tests in their article "Field Examination of Load Bearing Stone Structures." Articles such as these often discuss the details, success, and failure of the testing techniques used in individual research projects.

[11] Turkington, "Stone Durability," 25.

"Influence of Mechanical Anisotropic Behaviour to Tensile Strength on Decay Evolution of Marbles in Monuments" illustrates these complexities.[12]

Stone can be modeled as having no deterioration (an unlikely event), having a linear decrease in durability, asymptotic decrease in durability, or sudden and rapid deterioration over time. These models, illustrated in Figure 2.1, provide a way to analyze past, current or future rates of deterioration.

Models of Stone Deterioration Rates

Figure 2.1
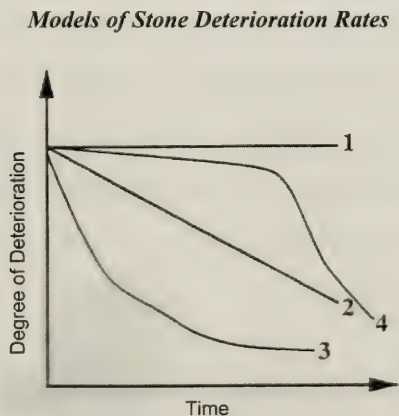
1 No deterioration, *2* linear deterioration, *3* asymptotic, and *4* sudden and rapid deterioration.

Reproduced from Turkington, "Stone Decay," in *Processes of Urban Decay, Proceedings of SWAPNET '95,* edited by Bernard J. Smith and Patricia A. Warke (London: Donhead Publishing, Ltd., 1996), 24.

---

[12] In *Conservation of Stone and Other Materials, Proceedings of the International RILEM/UNESCO Congress, Paris, 29 June-1 July 1993,* edited by M.-J. Thiel (London: E & FN Spon, 1993), 220-227.

Strength Profiles: Deteriorated Stone

In natural stones, deterioration can yield three distinct zones (from the exposed surface inward): the external zone on the exposed surface in which the weathered products are still attached to the stone body, the partially-weathered zone in which weathering mechanisms occur or are incipient, and the unweathered zone devoid of effects of weathering.[13] The external zone exhibits the highest amount of material loss and has the highest porosity.

Manifestation of these weathered zones brings about one of two strength profiles, depending on the form of deterioration. The *continuous strength profile* shows that strength gradually increases with depth; that is, as material is less deteriorated, it exhibits greater mechanical strength. The *discontinuous strength profile* is a result of scale formation whereby there is a sharp drop in strength at the scale interface. These profiles are shown in Figure 2.2.

These strength profiles illustrate that deterioration reduces the strength of stone. Thus, building stones lose their mechanical strength as they deteriorate. This loss of mechanical strength may be slow and insignificant to the structural performance of the stone in the short term. On the other hand, deterioration can have structural consequences if severe enough in the service life of the building.

---

[13] K. Gauri, D.J. Hagerty, and C.R. Ullrich, "Comparative Physical Properties of Weathered Impregnated and Unimpregnated Marble," *Engineering Geology* 6 (1972): 237. Their description was based on marble and provides a model for most other stones.

Strength Profiles of Deteriorated Stone
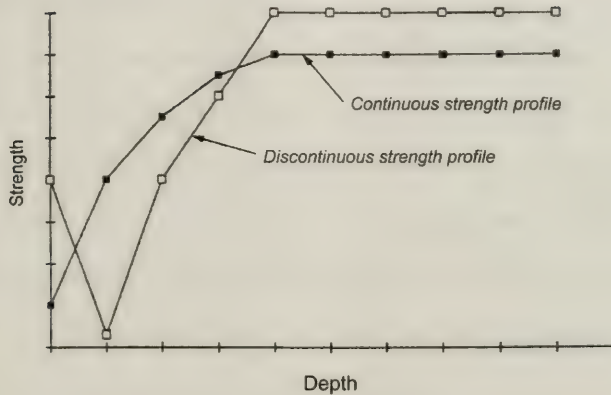
Figure 2.2

While some deterioration is said to contribute to the character and patina of a building, deterioration can interfere with the visual interpretation of the element or threaten the structural integrity of the building. For these reasons, an architect, engineer, and/or conservator are called to assess the extent and implications of deterioration, and propose ways of slowing or stopping further deterioration.

CHAPTER THREE: CONSERVATION TREATMENTS

Response to Deterioration

Stone, brick, terra cotta, concrete, and adobe are porous building materials subject to conservation treatments. Because the effects of consolidation treatments are the focus of this thesis, consolidants will be discussed in detail in this chapter. Stone and adobe are materials most often subject to consolidation, and much published research is available on treating these materials. For this thesis, research of consolidants was directed toward their application to natural stone. Stone is often used as a structural load-bearing material and is the subject of many building preservation projects.

"When confronted with decaying stonework, one's immediate instinct is to 'do something about it.'"[1] Indeed, this instinctual response brought about and advances the field of architectural conservation. The word *conservation* is derived from the Latin *conservare*, meaning to preserve or maintain. Strictly defined, conservation intends to preserve the status quo; but as Weber points out, conservation measures can and often do intend to regress or retard deterioration.[2] The purpose of conservation can be extended so far as to improve the appearance and/or structural performance of a material. To this end, conservation professionals are called to study a building and intervene in response to damage or deterioration.

---

[1] C.A. Price, *Stone Conservation: An Overview of Current Research* (Santa Monica, CA: The J. Paul Getty Trust, 1996), 13.

[2] Dr. Helmut Weber, "Stone Conservation - Planning and Execution," *ProSoCo Technical Bulletin 483-1* (Kansas City, KS: ProSoCo, Inc., n.d).

The response to building material deterioration can range from doing nothing (i.e, allowing deterioration to continue unimpeded) to intervening in the causes of deterioration, to applying a mitigating chemical treatment, to replacing the deteriorated stone.[3] Many factors influence the decision to intervene, maintain, or simply monitor deterioration: condition of the building, perceived value of the building, available technology, and cost of intervention.[4] The action taken should depend on how well the performance requirements of the material and of the intervention can be satisfied. Indeed, it is sometimes better to introduce no intervention measure rather than to introduce the wrong one, or one that proves ineffective or deleterious in the future. This principle of minimum intervention is advocated by conservation codes and guidelines such as the Venice Charter, the Burra Charter, and the *Secretary of the Interior's Standards for Rehabilitation.*[5]

Preventative conservation controls the causes of deterioration. Preventative conservation includes reducing or stopping access of surface water (by providing adequate drainage or flashing), controlling pollution which is eventually deposited on surfaces, managing visitation or occupancy of the building, controlling humidity levels inside or outside building, or erecting protective shelters around the building. Active conservation

---

[3] G. Sleater, "Development of Performance Criteria for the Selection of Stone Preservatives," in *Decay and Preservation of Stone, Engineering Geology Case Histories Number 11* (Boulder, CO: The Geological Society of America, 1978).

[4] Teutonico, "Group Report: How Can We Ensure the Responsible and Effective Use of Treatments (Cleaning, Consolidation, Protection)?", 304.

[5] The International Council of Monuments and Sites (ICOMOS) publishes the Venice and Burra Charters, as wells as other documents pertaining to the conservation of historic structures. The United States Secretary of the Interior provides the *Standards* as guidelines for work done to historic structures.

includes cleaning, desalinating, consolidating, and/or applying surface coatings to the material in response to or in anticipation of deterioration. Interventions such as mechanical pinning of fractured pieces or grouting void areas should be considered when the structural integrity of the building element is compromised. Replacing material – either wholesale or with Dutchmen repairs – should be considered when it is so badly decayed that doing nothing or applying a treatment are not effective means of preservation.

Before choosing any specific conservation intervention, the probable future course of the deterioration after intervention should be considered. If an architect, engineer, or conservator concentrates only on the immediate deterioration problem without taking a wider view of the structure or environment, he or she may worsen existing deterioration mechanisms or inadvertently inflict other deterioration mechanisms into the system. An intervention may begin as well-intentioned with a clearly defined objective, but in meeting that objective, it may create additional problems.[6]

An Overview of Stone Treatments

In light of minimum intervention strategies and of desire to retain as much historic material as possible in a structure, the application of chemical treatments to natural stone is a popular means of slowing its deterioration. Chemical treatment can avoid

---

[6] C.L. Searls, "Group Report: How Can We Diagnose the Condition of Stone Monuments and Arrive at Suitable Treatment Programs?" in *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage, (West Sussex, England: John Wiley & Sons Ltd, 1997), 210-211

mismatches in color or texture that can occur when replacing stone. Treatments – also called stone preservatives – attempt to prolong the life of a stone, either by retarding the deterioration processes or by restoring the physical integrity of the stone.[7]

Brief History of Treatments

Chemical compounds have been used for over two thousand years in an effort to protect and conserve stone. A mixture of molten wax and oil was used on marble statues in the first century BC. Vitruvius described rubbing solid wax on warmed surfaces or even immersing stone in molten wax.[8] Mixtures of wax plus resin, sandrac, nut oil, incense and potash alum were used in the fourteenth and sixteenth centuries.[9] In the middle ages, painting was a means of both decorating and protecting stone. In fact, the Public Palace of Louvain was painted with oil in 1567 because the stone was "easily alterable due to frost."[10] Up until the nineteenth century, it was common to give stone buildings a protective coat of plaster, distemper or paint.[11] In the first part of the nineteenth century, emphasis shifted from protecting stone with water repellents toward stone consolidation.[12] Thomas Egleston at the Columbia School of Mines began experiments

[7] Sleater, "Development of Performance Criteria for the Selection of Stone Preservatives," 65.

[8] Clifford A. Price, "Stone - Decay and Preservation," in *The Association for Preservation Technology Masonry Conservation and Cleaning Handbook* (The Association for Preservation Technology, 1984), 131.

[9] Alan H. Spry, "Effectiveness of Preservatives and Surface Coatings in the Protection of Masonry," *ICCM Bulletin* 13: 65.

[10] A. Elena Charola, "Water-Repellent Treatments for Building Stones: A Practical Overview," *APT Bulletin* 26 (1995): 10.

[11] Peter J. Koblischek, "Polymers in the Renovation of Buildings Constructed of Natural Stone in the Mediterranean Basin," in *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 3rd International* Symposium (Venice: Soprintendenza ai Beni Artisitci e Storici di Venezia, 1994), 850.

[12] Charola, "Water-Repellent Treatments for Building Stones," 10.

in the 1860s involving brush application of boiled oil.[13] In 1861 Hofman called attention to the potential of tetraethylorthosilicate (silicic ester) for stone treatment.[14] In 1863 J.C. Coombe was granted the first American patent for a masonry treatment: application of fluorosilicic acid, or alkaline salts followed by fluorosilicic acid, to stone. In the 1880s, Egleston developed formulations of paraffin, oil, and sulfur to be applied hot to stone; other researchers recommended variations of this formula. Although somewhat effective, wax and paraffin formulations were observed to exacerbate soiling of the stone. In the early twentieth century, Noel Heaton, a British chemist, pioneered the study of stone conservation; he introduced the points of treatment penetration, permeability of treatment to water vapor, and color changes of the stone due to treatment.

The growth of the organic chemistry field in the early 1900s led to production of new conservation chemicals. Vinyl resins were first manufactured in the mid- to late-1920s. After World War II, silicones developed by Dow Corning for airplanes were used in conservation applications. Epoxies were developed in the 1950s, and Domaslowski reported the first successful consolidation of deteriorated stone with epoxy in 1967. Throughout the second half of the twentieth century, various synthetic chemicals were introduced and tested, including methyl methacrylates, ethyl silicates, and acrylics. Recent environmental concerns have brought about research into water-born treatments.

---

[13] Norman Weiss, "Chemical Treatments for Masonry: An American History," *APT Bulletin* 26 (1995): 10. Unless otherwise noted, information on the history of treatments comes from this article.

[14] A.P. Laurie patented its use in 1926. From S.Z. Lewin, "The Current State of the Art in the Use of Synthetic Materials For Stone Conservation: Inorganic and Metal-Organic Compounds," in *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration* (Paris: UNESCO, 1988), 293.

The list of chemical treatments available today is long, with each product fulfilling a stated objective or suited to one type of substrate. Much research, development and testing of these products has been done during the past thirty years. The bibliography at the end of this thesis includes only a fraction of the published books and articles available on the subject of stone treatment.

Types of Treatments

Stone treatments are classified into four main types: cleaning, joining or gap-filling, consolidating, and protecting. One conservation project may include one or more of these treatments; the selection and sequence of treatment depends on the state of the stone and the desired performance of the treatment.[15] Furthermore, selection considerations for treatment type or specific product should include the objective of the treatment, the historic preservation guidelines applicable to the building, the composition and characteristics of the stone, the performance of the treatment on the stone, and the application method and its practicality on site.[16] An overview of each type follows.

Cleaning Treatments

Cleaning concerns the surface appearance of stone. Removal of dirt, oils, pollutants, salts and salt efflorescence, and/or biological growth can be a conservation treatment in itself

---

[15] M. Laurenzi Tabasso, "Conservation Treatments of Stone," in *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper (Paris: UNESCO, 1988), 271.

[16] Carolyn Searls, and David P. Wessel, "Guidelines for Consolidants," *APT Bulletin* 26 (1995): 43.

25

or as a first step prior to further treatment. Surface contaminants can enhance deterioration of stone or cause failure of any applied treatments.

Cleaning stone can be done by washing with water, steam, and/or chemicals; or by removing unwanted material with air abrasives, poultices, or mechanical means (brushes, chisels or lasers). All of these means can be effective, but inappropriate application of them can exacerbate future deterioration in some cases. Efficacy and harmlessness (to the stone, environment and people) are two key requirements that every cleaning method must fulfill.[17]

Joining or Gap-Filling Treatments

Delaminated or detached pieces of stone can be reattached with mortar, adhesives, or epoxies. Large cracks, voids, or surface losses can be filled with either mortar or epoxy, depending on the exposure environment and need for structural support. Mortar is often more compatible to the stone in terms of its mechanical properties, is relatively inexpensive, and is easy to mix and apply. Epoxy is comprised of a resin plus a hardener, which are mixed together then applied by injection, brushing, or spraying. Epoxies have good adhesive characteristics and can penetrate into porous substrates to fill deterioration losses. Epoxies are durable and exhibit excellent mechanical strength; however, they can often lead to discoloration of the stone substrate if applied inappropriately. Epoxies can

---

[17] Jose Delgado Rodrigues, and Elda De Castro, "Some Remarks on the Efficacy and Harmfulness of Stone Cleaning," in *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 1st International Symposium, Bari, 7-10 June 1989,* edited by F. Zezza (Brescia, Italy: Grafo, 1990), 491.

also be used as consolidants (see below), as demonstrated by Gauri and described by Selwitz.[18] Epoxies imbue increased mechanical strength to substrates, but deleterious effects of composite action between them and their substrates should be considered.

Consolidating Treatments

Consolidation is the process of applying natural (organic or inorganic) substances, or natural or synthetic polymers or prepolymers into deteriorated stone – usually stone exhibiting flaking or sugaring as described in Chapter Two. The purposes of surface consolidation are to improve the cohesion of mineral constituents and achieve adhesion between deteriorated and sound zones. Fully-impregnated consolidants (such as epoxies) increase the mechanical strength of deteriorated stone, and some applications of partially-penetrating consolidants can also increase stone's mechanical strength (see Table 5.1). A consolidant reestablishes the bond between particles lost through weathering and deterioration, and aims to reduce the rate of deterioration. A secondary effect of consolidation is reduced porosity of the stone; in fact, preferred are consolidants that reduce penetration of liquid water while still allowing water vapor permeability.[19]

Consolidants are classified as either organic or inorganic, with several types of materials falling into each class.

> *Organic Consolidants*
> - Natural: oils, waxes, paraffin, gelatin, resins

---

[18] Use of epoxy is described thoroughly by Selwitz, *Epoxy Resins in Stone Conservation* (Santa Monica, CA: The J. Paul Getty Trust, 1992).
[19] M. Laurenzi Tabasso, "Conservation Treatments of Stone," 281.

- Synthetic: polyethylene, poly(vinyl) products, acrylics (such as methyl methacrylate and butyl methacrylate), alkoxysilanes (such as ethyl silicate, also called tetraethoxysilane), alkyl alkoxysilanes (such as triethoxy-methylsilane and trimethoxy-methylsilane), epoxies

Inorganic Consolidants
- Alkaline earth hydroxides: lime wash and barium hydroxide (baryata) wash
- Alkali silicates: dispersions of silica in sodium hydroxide or soluble alkali silicates
- Silicates: fluorosilicates
- Acids: hydrofluoric, phosphoric
- Stearates: zinc or aluminum

Consolidation without resolving the underlying mechanism of deterioration can cause the treatment to fail or to even accelerate the rate of deterioration. Thus, consolidation should be done only after unsuccessful attempts to remove or modify deterioration mechanisms have been carried out.[20] Consolidation should prolong the service life of the stone, but as Torraca notes, "Usually consolidation does not stop deterioration, even if it has a favorable effect on the deterioration rate."[21] Application of a consolidant is justified when:

- stone is deteriorating at an unacceptable and quantifiable way,
- the causes of deterioration have been properly identified and understood,
- there are no practical means of slowing deterioration by modifying the environment,
- all possible alternative interventions have been explored and refuted,
- the properties, constituents and application technique of the consolidant are known, understood, and can be executed,
- full documentation of the structure before treatment is carried out.[22]

[20] American Society for Testing and Materials, "Guidelines for the Selection and Use of Stone Consolidants, Draft No. 11" (Philadelphia, PA: ASTM, September 1998), section 4.5.

[21] Giorgio Torraca, "General Philosophy of Stone Conservation," in *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper (Paris: UNESCO, 1988), 248.

[22] John & Nicola Ashurst, *Practical Building Conservation, English Heritage Technical Handbook Volume 1: Stone Masonry* (New York: Halstead Press, 1988), 89.

Consolidants should not substitute for grouts, joining, or gap-filling treatments. They should not be applied to zones of major moisture movement, to wet surfaces, or to surfaces with high concentration of soluble salts.[23] Consolidants will be discussed in more detail later in this chapter.

Protective Treatments

Because water is a major deteriorating agent, protective treatments attempt to shed water to some extent. Protective treatment involves the application of a waterproofing or water repellent product to the exposed surface of the stone or deep within the pore structure of the stone. The treatment must reduce the penetration of water and any material it carries.[24] Protective treatments can yield the stone totally impermeable to water (waterproofing), or impermeable only to liquid water while allowing passage of water vapor (water repelling). Water repellent coatings are usually more suited to existing stone construction, since they allow any moisture trapped in the stone to migrate to the stone surface. Charola discusses the chemistry of water repellent compounds in detail.[25] Some consolidants – triethoxymethylsilane, trimethoxymethylsilane, and ethyl silicates to some extent – impart water repellency by virtue of their methyl or silicon resin groups.

Lukaszewics advises that any consolidation treatment should be followed by application of a water repellent to protect the stone against the action of liquid water.[26] This advice

---

[23] Clifford A. Price, "Stone - Decay and Preservation," 91.

[24] M. Laurenzi Tabasso, "Conservation Treatments of Stone," 288.

[25] Charola, "Water-Repellent Treatments for Building Stones: A Practical Overview," 10-17.

[26] J.W. Lukaszewics, "Application of Silicone Products in the Conservation of Volcanic Tuffs," in *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990* (Rome: ICCROM, 1994), 192.

should not be followed blindly; rather, the effect of combining consolidating and water repellent treatments in tandem should be confirmed prior to application on a specific stone structure. Recent research has shown that silicone resins show a remarkable drop in efficiency after a period of some years: they are able to prevent capillary uptake of water for only a short period of time.[27] Mavrov suggests flaws in protective coatings (specifically, the number of cracks) could be decreased by covering them with repeated layers of film.[28] Repeated application of protective treatments, however, may lead to changes in the surface appearance (changes in the color or reflectance), or even to changes in the physical-mechanical properties of the stone (formation of a rigid surface zone). Thus, some types of coatings or inappropriate application of coatings may cause greater damage to stone. Although water repellent treatments have many benefits, their disadvantageous effects can outweigh these. Wendler suggests protective treatment of stone when:

- capillary absorbency is high,
- there are reactive components (metal reinforcement and the like) in the structure,
- it is possible to retreat the stone without altering the mechanical properties of the treated zone,
- moisture pathways from the ground or other sources are absent,
- no soluble or hygroscopic salts are in the stone.[29]

With the preceding discussion of treatments, it should be noted that no one treatment exists that universally solves or slows all deterioration problems. Furthermore, when one

---

[27] Wendler, "Materials and Approaches for the Conservation of Stone," 182.
[28] George Mavrov, "Aging of Silicone Resins," *Studies in Conservation* 28 (1983): 177.
[29] Wendler, "Materials and Approaches for the Conservation of Stone," 188.

type of treatment, or one specific product, is effective on one type of stone, it may not be effective or suited to another type of stone. In fact, properties of some treatments may be mutually exclusive of each other.

A Closer Look at Consolidants

Because of their proven ability to change – to increase or even decrease – the mechanical properties of stone after application, this thesis focuses on consolidating treatments. Weber suggests the following criteria for identifying a good consolidating product: it forms a new weather-resistant binder; allows good penetration depth, does not form a surface crust, but instead allows even consolidation through depth; does not form harmful by-products (specifically, salts); does not impart surface color change; does not appreciably change physical characteristics (water vapor transmission, thermal expansion properties, and the like) of the stone; and reduces water absorption of the stone (to slow rate of further deterioration).[30]

Physical Models of Consolidation

The strength of stone is ultimately defined by the strength of its grain structure. This strength is built up by three different mechanisms: cohesion due to real mineral bridges between grains, cohesion of minerals due to electrostatic forces and water films, and cohesion due to mechanical interlocking of minerals. A loss of strength or cohesion of

---

[30] Dr. Helmut Weber, "Conservation and Restoration of Natural Stone in Europe," *APT Bulletin* 17 (1985): 16.

grains means that these forces have weakened or have been lost.[31] Consolidants either fill the grain structure's pores or deposit a film over the grains and micropores to reinstate the physical-mechanical binding of stone. In general, silica gels bridge between grains, and artificial resins form films over the grains.[32] Surface-applied treatments may consolidate only an outer zone of deteriorated material, adhering it to an underlying solid core of stone. Fully-impregnating treatments rebind grains of wholly deteriorated stone and in effect reconstitute the stone.

Observation with scanning electron microscopy (SEM) and thin section microscopy helps researchers understand how consolidants physically interact with their substrates to effectively rebind their grains. Saleh, et al., observed sandstone treated with Paraloid B-72 (an acrylic resin): a polymer network was seen, as was a fine membrane of spongy appearance coating the sandstone grains. They also observed the inability of B-72 to penetrate the internal structure of the stone because of the chemical's high viscosity. Because the B-72 only partially dissolved in its solvent, precipitant B-72 partially filled the pores between sandstone grains. In sandstone treated with methyl trimethoxysilane (MTMOS), they saw precipitated polymer "nodules." In sandstone treated with B-72 dissolved in MTMOS, they observed the grains coated with consolidant such that the boundaries between grains were obscured. With this treatment they also saw a network

[31] H. R. Sasse, and R. Snethlage, "Methods for the Evaluation of Stone Conservation Treatments," in *Dahlem Workshop Reports: Saving Our Architectural Heritage, The Conservation of Historic Stone Structures, March 3-8, 1996* (Berlin: London: John Wiley & Sons, 1996), 235.

[32] H. Rainer Sasse, and R. Snethlage, "Evaluation of Stone Consolidation Treatments," *Science and Technology for Cultural Heritage* 5 (1996): 87.

structure of consolidant dispersed between grains and through the pores. In a specimen

treated with a siloxane, they observed a thin layer of polymer on the grains, but the grain

boundaries were still visible. Sandstone treated with Wacker OH (a tetraethoxysilane)

showed the spread of a network structure of the polymer on and between the grains, and

in depth between the pores.[33] Hristova and Todorov saw a similar network structure on a

sandy limestone treated with Wacker OH: the consolidant was on the surface of the

grains, in the contact zone between grains, and in the pores.[34]

De Witte, Charola, and Sherryl investigated Belgian limestone treated with methyl

propylsiloxane, ethyl silicate, polyurethane, or fluosilicate, with all specimen etched with

dilute acid before SEM observation. In those specimen treated with methyl

propylsiloxane, they observed a film-like deposit. In those treated with ethyl silicate,

they saw a very fine, spongy residue that occurred in discrete units (illustrating that ethyl

silicate polymerizes around a catalyst nucleus). In those treated with polyurethane, they

saw a spongy residue like that of the ethyl silicate. In those treated with fluosilicate, they

saw a calcium fluoride deposit that formed on the calcite surface; however, the residue

left behind did not seem to be attached to the calcite grains, but was instead deposited

---

[33] Saleh A. Saleh, F.M. Helmi, M.M. Kamal, and A.-F. E. El-Banna, "Study and Consolidation of Sandstone: Temple of Karnak, Luxor, Egypt," *Studies in Conservation* 37 (1991): 98-99.

[34] Julia Hristova, and V. Todorov, "Consolidation Effect of Wacker-Silicones on the Properties of Sandy Limestone," in *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer (Berlin: moller druck und verlag gmbh, 1996), 1198.

interstitially.[35] In another SEM study of treated limestone, Charola, et al., observed that MTMOS did not provide a completely uniform film over the mineral particles. Dri-Film 104 formed a fairly uniform and heavy coating on the stone particles in some areas, while in other areas they saw a thinner film; furthermore, the resin was only deposited on the particles but did not form an evident attachment between particles. They commented that magnification shows that treatments may not form an absolutely uniform coating on stone particles even when impregnation is homogeneous in the stone.[36]

With these and other studies, it is noted that some treatments may not be effective on some types of stones. For example, ethyl silicate may not uniformly coat limestone (calcite) grains as it does on sandstone (silicate) grains. Goins, Wheeler, and Wypyski applied Conservare OH (identical to Wacker OH) or MTMOS to both quartz and calcite crystals, in an attempt to simulate application on sandstone and limestone. On both types of crystals, the OH formed similar types of films: brittle with no apparent adhesion to the crystal surface. The MTMOS formed no film on either the quartz or calcite; instead, small discrete particles were seen loosely attached in patch areas over the surfaces. They observed, however, that while there may not be chemical bonding between the quartz and the MTMOS gels, the gels are not repelled or interfered with by quartz so that the quartz is consolidated. Interestingly, they saw that undiluted amino alkoxysilanes formed

---

[35] E. De Witte, A.E. Charola, and R.P. Sherryl, "Preliminary Tests on Commercial Stone Consolidants." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix (Lausanne, Switzerland: Presses Polytechniques Romandes, 1985), 715.

[36] A.E. Charola, A.E., R. Rossi-Manaresi, R.J. Koestler, G.E. Wheeler, and A. Tucci, "SEM Examination of Limestones Treated with Silanes or Prepolymerized Silicone Resin in Solution," in *Preprints of the IIC Congress "Adhesives and Consolidants,"* (Paris, 1984), 182-184.

adherent, cohesive, and somewhat flexible films that had affinity for both quartz and calcite.[37]

From observations like these, and after considering the aim of a consolidating treatment, Sasse and collaborators have developed the "supporting corset model" (also called the Aachen Concept of Stone Preservation) to describe the properties of effective consolidants. In this model, a protecting and strengthening polymer microlayer of consolidant coats the internal pore surfaces of the stone. Grains are expected to be evenly coated by the microlayer so that they are relinked to each other by "grain bridges." As a result of expected mechanical properties, the polymer microlayer displays the function of a supporting corset. The microlayer should be of thickness in the nanometer to micrometer range. Figure 3.1 illustrates this model of consolidation.

Ideally, the microlayer itself should be water repellent and water resistant, impermeable against water and water vapor, resistant against chemical and biological agents, and should show elastic deformation (see Chapter Four) through a wide range of temperatures. The treated stone, however, should show no significant changes in water vapor transmission since the large capillary pores in the bulk volume of the stone remain

---

[37] E.S. Goins, G.S. Wheeler, and M.T. Wypyski, "Alkoxysilane Film Formation on Quartz and Calcite Crystal Surfaces," in *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996*, edited by Josef Riederer (Berlin: moller druck und verlag gmbh, 1996), 1255-1264.
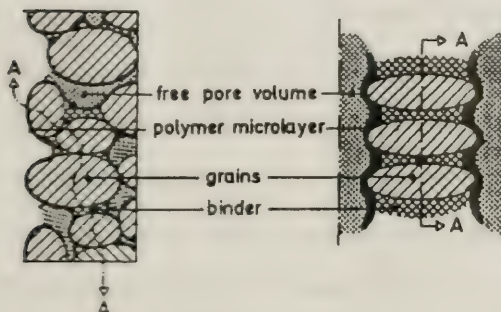
Figure 3.1

Reproduced from H.R. Sasse, D. Honsinger, and M. Puterman, "The Aachen Concept: A New Technology in Stone Impregnation," in *1ˢᵗ International Colloquium Role of Chemistry in Archaeology, 15-18 November 1991* (Hyderabad, India: Birla Institute of Sceintific Research, 1991), 88.

open. The microlayer of consolidant differs from a water repellent because it coats all inner surfaces of the stone.[38] Current work is developing microlayer-forming polymers.

Appropriateness, Effectiveness and Durability of Consolidants

The appropriateness and effectiveness of a consolidant should be determined by comparing physical, chemical, and mechanical parameters of the treated stone to those of the untreated stone (both freshly quarried and deteriorated). In addition, the durability or service life of the treated stone, practicality of application, and environmental health/safety effects should also be considered. "Appropriateness," "effectiveness," and "durability" can be somewhat subjective terms. RILEM 25 PEM (Protection et Erosion

---

[38] H.R. Sasse, "A New Chemical and Engineering Approach for Development and Optimization of Stone Protecting Materials," in *Science, Technology and European Cultural Heritage, Proceedings of the European Symposium, Bologna, Italy, 13-16 June 1989* (London: Butterworth-Heinemann Ltd., 1991). Others working with Sasse include Honsinger and Puterman (see bibliography).

des Monuments), NORMAL (Normativa Manufatti Lapidei), ASTM (American Society for Testing and Materials), the NBS (National Bureau of Standards), DIN (Deutsche Industrie-Normen), as well as independent researchers have published guidelines and/or standards for testing materials treated with consolidants. The specific tests used should always be chosen based on the objective of the treatment and its intended outcome; furthermore, it is wise to determine if treatment yields any *unintended* outcomes. NORMAL 20/85 suggests determining the change in weight or bulk density, change in color, change in specific surface, water absorption rate (by total immersion, capillarity, and low-pressure), water evaporation rate, pore-size distribution, water vapor permeability, depth of penetration, mechanical strength, and abrasion resistance.[39] Others suggest testing the amount of consolidant absorbed, the change in pore characteristics, and surface hardness. Mechanical tests have been included in some studies, as will be discussed in more detail in Chapter Five.

John and Nicola Ashurst suggest that shallow or pore-blocking treatments are "useless or worse than useless – they exacerbate the condition they are trying to improve and make better and later treatments difficult or impossible."[40] This statement in explored quantitatively in Chapter Five. Careful testing and analysis may show that a consolidant is an inappropriate treatment.

---

[39] A. Elena Charola, "Laboratory Tests and Evaluation of Proposed Masonry Treatments," *APT Bulletin* 26 (1995): 37.
[40] *Practical Building Conservation*, 90.

Treatments are considered effective when they slow the rate of deterioration and increase durability.[41] Efficacy of treatment depends on the characteristics of substrate, the product used, the method of application, and environmental exposure. Suggested "effectiveness indices" have been developed to judge the treatment to relate before-and-after values given by tests. Sleater gives qualitative requirements of treatments.[42] Sasse and Snethlage attempt to quantify the requirements of treated material.[43] It should be noted that some characterization tests (porosity, pore size distribution, color, depth of penetration) do not give any direct indication of the treatment's effectiveness; but rather, conservators use these parameters for their perceived value on the effect of treatment and its durability.[44] Tests provide valuable bases for choosing and evaluating treatments; researching case studies of similar applications and acquiring field experience with treatments can often be real judges of the treatment's effectiveness. Application and evaluation of the treatment on the actual structure determines the product's effectiveness in the long run.

If tests prove the treatment to be effective and if the performance goals of the treatment are met, trial applications in a laboratory and/or *in situ* should be carried out to determine

---

[41] Teutonico, "Group Report: How Can We Ensure the Responsible and Effective Use of Treatments (Cleaning, Consolidation, Protection)?" 296.

[42] In "Stone Preservatives: Methods of Laboratory Testing and Preliminary Performance Criteria," *NBS Technical Note 941* (Washington, DC: U.S. Department of Commerce, National Bureau of Standards, May 1977).

[43] In "Evaluation of Stone Consolidation Treatments;" and "Methods for the Evaluation of Stone Conservation Treatments."

[44] C.A. Price, "Report on Session VI (Weathering)," in *ICCROM International Colloquium: Methods of Evaluating Products for the Conservation of Porous Building Materials in Monuments (In Science and Technology in Cultural Heritage), 19-21 June 1995 (*Rome, 1995), 107.

the durability of the treatment. Natural weathering is the only true test of a product's durability – the product is not effective if it is not durable. However, natural weathering takes time, and a treated stone will weather differently in different environments and locations. Although treatments applied twenty years or so ago are now being evaluated for effectiveness and durability, most applications require more immediate measures of durability and performance. Artificial and accelerated weathering tests of lab specimen are often used to determine the durability of a consolidant prior its application on a building. Freeze-thaw tests, salt crystallization tests, wet-dry cycling, acid cycling, and ultraviolet radiation cycling help predict the service life of both untreated and treated stone. Weight loss, surface changes, and pore changes are generally recorded during these tests. Artificial and accelerated weathering tests do have their pitfalls; for example, the tests may not simulate real-life situations, such as large-scale architectural and construction details of the structure.

After applying the treatment to the actual building, low-destructive and non-destructive tests can aid in monitoring the performance of the treatment. Ultrasonic testing provides information about the density and mechanical properties of a material both before and after treatment. Pulse velocity methods can determine depths of untreated and treated material, which could be used to track deterioration and the durability of the treatment over time. Surface hardness tests can also be used to monitor durability over time. All non-destructive methods require some type of calibration, which may limit their applicability to some situations.

One should consider the fact that consolidation is an irreversible process – at least until the consolidant and its substrate weather away. Many in the conservation field suggest that a treatment should be reversible; but because of chemical processes involved in consolidation and because of often deeply-penetrating application, it is usually impossible to remove all traces of treatment. Many now suggest that consolidation should be a *retreatable* treatment, anticipating the event the initial treatment fails or is replaced by better products.

Strength Profiles: Treated Stone

Mihaly Zador states that conservation problems belong to three basic types: those stones needing no strengthening (only protection), those needing strengthening to a minor depth (less than 10mm), and those needing deep strengthening (30-40mm).[45] Several researchers indicate that acceptable consolidation yields a strength-versus-depth profile which is nearly that of undeteriorated stone – not weaker, not stronger.[46] Although actual measurement of this profile is difficult, Weber and Zinsmeister have depicted schematic strength profiles of deteriorated stone before and after treatment, as shown in Figure 3.2.

---

[45] In "Activity of the RILEM 25 PEM - ICOMOS Working Group: Its Utilization and Instructions in Monuments Restoration," *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments* (Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978), 6.

[46] M. Boos, J. Grobe, G. Hilbert, and J.Mler-Rochholz, "Modified Elastic Silicic-Acid Ester Applied on Natural Stone and Tests of Their Efficiency," in *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996* (Berlin: moller druck und verlag gmbh, 1996), 1179; and Koblischek, "Polymers in the Renovation of Buildings Constructed of Natural Stone in the Mediterranean Basin," 849.

**Strength Profiles of Deteriorated Stone
Before and After Treatment**
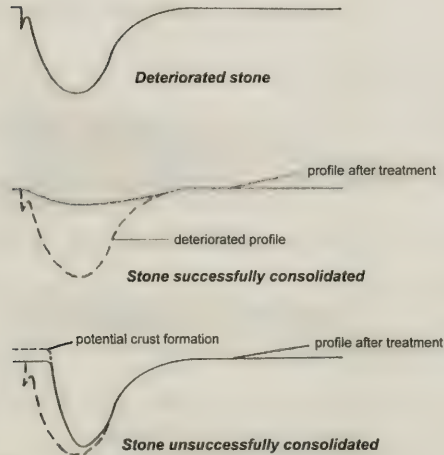
Deteriorated stone

profile after treatment

deteriorated profile

Stone successfully consolidated

potential crust formation

profile after treatment

Stone unsuccessfully consolidated

Figure 3.2

Their model for "stone unsuccessfully consolidated" shows little or no strengthening of the deteriorated zone and the formation of an over-strengthened surface zone. The model for "stone successfully consolidated" is ideal, but may not be achieved in reality. Surface-applied consolidants rely on capillary action of low-viscosity treatments to penetrate and consolidate substrates whose permeability most likely varies within its body. It is unlikely that the consolidant uniformly or linearly penetrates porous building materials. As the models show, strength after treatment will vary with penetration depth of the treatment. However, the strength profile for surface-applied and partially-penetrating consolidants may never be as flat as depicted by the model.

CHAPTER FOUR: STRUCTURAL MECHANICS

Complexity of Determining Material Properties

When analyzing a structure built of any material, an engineer must know the structure's geometry, its construction technique (number of wythes in the wall, joint thickness, and mortar type), and the physical and mechanical properties of the material itself and of the composite construction (for example, stones plus mortar joints). As discussed in Chapter Two, building materials deteriorate – a process that can result in the loss of material from exposed surfaces. Loss can lead to changes in physical and mechanical properties of the material, including reductions in density, cross-sectional area, and even mechanical strength. Treating deteriorated structural stone with consolidants, as discussed in Chapter Three, can slow material loss, restore mechanical strength, and even increase mechanical strength beyond original values.

The structural behavior of any material – treated or untreated – can be complex to analyze. As Table 2.1 illustrated, the strength parameters of untreated stone can vary from one stone type to another. Furthermore, the properties of one type of stone can depend on the source quarry (geologic location), and the strength of one stone specimen can depend on the orientation of its mineral crystals or bedding planes to applied loads. Properties of brick and terra cotta depend on the source clays, additives, and firing temperature. The strength of concrete depends primarily on the mix proportions and curing environment, both of which can vary from project to project or even within one building. When a conservation treatment is applied to any porous material, its properties

may be altered through the depth of the treatment but not in the untreated zone, such that determination of properties and structural performance of the element as a whole becomes even more complex. Without testing (either destructively or nondestructively) it is difficult to accurately predict the material properties of fresh, deteriorated, or treated material.

This chapter presents the basic theories and equations used in structural mechanics and analysis of both pure and composite materials. This overview will illustrate those mechanical properties needed to complete structural analysis and how the magnitude of those properties and subsequent structural responses can determine the success or failure of the material and structure. Particular applications will focus on the mechanics of treated and untreated stone and concrete, as they are the materials of focus in this thesis.

Types of Materials

Materials used in structural elements can be either pure or composite; that is, they can be described as homogenous or heterogeneous. The distinction between pure and composite materials can be confusing and depends on the scale (microscopic or macroscopic) with which the material is examined.

Pure Materials

A *pure* material is defined as one that is "free from anything adulterates or taints;" one that is "unmixed."[1] Except in highly controlled laboratory environments, it is unlikely that any material is actually "pure." Foreign atoms often interrupt the atomic structure of metals; wood grows with a combination of heartwood and sapwood, comprised of both "clear" wood and knots; and stone is often an agglomeration of several types of minerals bound by a cementing matrix. Thus, on a microscopic scale, almost all materials are not pure or homogenous, but are instead heterogeneous. This microscopic impurity certainly affects the physical, chemical and mechanical properties of the material. There exists a scale, however, at which the properties of the constituents can be negligible or discounted and are averaged such that the material is said to be effectively homogeneous or pure. This is done because gross property performance is not affected by micro-structure idiosyncrasies.

Composite Materials

Jean-Marie Berthelot defines a *composite* material as "made of two or more different parts."[2] Robert Jones defines a composite as "two or more materials combined on a macroscopic scale to form a useful third material."[3] These definitions differ in that Berthelot does not provide for a third material, but rather, that the two materials are

---

[1] *Webster's New World Dictionary* (New York: Warner Books, 1990).
[2] *Composite Materials: Mechanical Behavior and Structural Analysis* (New York: Springer-Verlag, 1990), 3.
[3] *Mechanics of Composite Materials* (Philadelphia, PA: Taylor & Francis, Inc., 1999), 2. Both Jones' and Berthelot's books provide background information about composite materials.

blended homogeneously. The purpose of using a composite material is to exploit the best qualities of the constituent materials, often producing qualities that none of the constituent materials possess. The properties of composite materials, then, result from the properties of the constituent materials, their geometric and volumetric distribution, and their interactions. Increased strength, stiffness, corrosion/deterioration resistance, thermal insulation/conductivity, and decreased weight are some advantages of using composite materials. An interesting activity of this thesis will be to determine if there are any *disadvantages* to using composites, particularly in the case producing composite porous materials via partial-depth consolidation.

There are four common types of composite materials: fibrous (fibers in a matrix), particulate (particles in a matrix), laminated (layers bound together), and combinations of these. Reinforced concrete is a traditional "fiber" composite, in which a steel bar is bound in a matrix of concrete. The concrete is strong in compression, the steel is strong in tension, and the resulting composite is strong in both tension and compression. Straw-reinforced adobe or horsehair-reinforced plaster works on the same principle. Concrete itself can be described as a particulate composite, with its constituent aggregate bound in the cement matrix. Consolidated stone could be described as a particulate composite if the treatment penetrates the entire cross-section of the element, or as a laminate composite if a layer of consolidated stone is bound to the remaining untreated stone. One can examine a composite material at the microscopic scale to study the interaction of its constituent materials and how they perform individually, or at the macroscopic scale whereby the whole is considered homogeneous to determine its overall performance.

One of the most important factors determining the mechanical characteristics of a composite material is the relative proportion of reinforcement (fibers or particles) in the matrix. This property is commonly expressed as a volume fraction of matrix (V_m) or fiber/particle ($V_{f,p}$) to the material as a whole, where $V_m + V_{f,p} = 1$.

Material Type Definitions for This Thesis

Berthelot suggests that at the engineering level the characteristic of heterogeneity appears whenever the physical or mechanical properties of a material are a function of point location within the material.[4] For the purpose of this thesis, however, any one material type – stone, consolidant, or concrete – will be considered as a pure material. Constituents of pure materials – mineral grains, consolidant solvent, cement matrix, or aggregate – will be discounted unless specifically stated. Any combination of pure materials – stone + consolidant, or concrete + consolidant – will be classified as a composite material.

Goins, Wheeler and Fleming state that a consolidated stone could be a type of composite material.[5] They suggest that there can be adhesion between the substrate and consolidant, which could increase mechanical strength, as in a typical laminate composite. If there is no adhesion between the two phases, any increase in strength could be due to the filling effect of the consolidant gel, as in a particulate or fiber composite.

---

[4] *Composite Materials: Mechanical Behavior and Structural Analysis,* 149.
[5] E.S. Goins, G.S. Wheeler, and S.A. Fleming, "The Influence of Reaction Parameters on the Effectiveness of Tetraethoxysilane-Based Stone Consolidants: Solvent Effects," in *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995* (Rome: ICCROM, 1995), 260.

Mechanics of Pure Materials

The analysis and design of an entire structural assembly involves the determination of stresses and deformations of its individual components. Stress and deformation are determined by the response of the constituent material – either pure or composite – to applied loads.[6]

Applied loads cause deformation (δ) – a change in the original length (L) of an element. Strain (ε) is deformation per original unit length (gage length) of the element:

$$\delta = \Delta L \quad \text{and} \quad \varepsilon = \frac{\Delta L}{L}.$$

Force (F) applied over an area (A) is called the stress (σ) felt by that section:

$$\sigma = \frac{F}{A}.$$

Axial stress applied perpendicularly to an element can be in tension (pulling) or in compression (squeezing). When axial stress is applied centrically to the specimen, the resulting stress is distributed uniformly over the specimen's cross-section. When transverse forces are applied to a member, shearing stresses (τ) result. Shear stress is not uniform across an area, but instead varies linearly over the section.

Applying and removing loads, and noting resulting deformations, is one way to determine materials' mechanical properties. Plotting stress versus strain graphically after such

---

[6] An excellent introductory text for mechanics of materials and structural analysis is: Ferdinand P. Beer, and E. Russell Johnson, *Mechanics of Materials*, 2nd ed. (New York: McGraw-Hill, Inc., 1992). Unless otherwise noted, information for this chapter was taken from this book.

testing produces a stress-strain diagram. Deformation will occur as load is applied, and it will disappear after load is removed from a so-called elastic material. Elastic materials exhibit a linear relationship between stress and strain up to the yield stress (σ_y). The proportional constant of this relationship is called the modulus of elasticity (E). E (in units of stress) describes the stiffness of a material and its ability to resist deformation. The linear relationship between stress and strain is expressed by Hooke's Law:

$$\sigma = E\varepsilon.$$

The modulus of elasticity can be determined by mathematical calculation if σ and ε are known, or by graphical interpretation as the slope of the linear portion of the σ-ε diagram.

During loading, deformation can be measured or calculated using by the equation

$$\delta = \frac{FL}{AE}.$$

After loading an elastic material beyond yield stress, stress and strain are no longer related linearly, and large strains occur with small increases in stress. This phenomenon is called plastic deformation, and is a result of whole planes of atoms translating relative to adjacent planes. These planes do not translate in the opposite direction during unloading, resulting in "permanent set" when the load is removed (that is, ε does not return to zero).[7]

---

[7] Joseph B. Walsh, "Deformation and Fracture of Rock," *Conservation of Historic Stone Buildings and Monuments* (Washington, DC: National Academy Press, 1982), 89.

Depending on their response to loading, materials can be classified into the following categories:

- *Linear elastic.* Resists both tension and compression linearly to strain.
- *Nonlinear elastic.* Resists compression but not tension.
- *Elasto-plastic/perfectly plastic.* Yields under load and undergoes permanent strain.[8]

Stress-strain diagrams are useful in describing a material as ductile or brittle: brittle materials usually rupture or fail without noticeable change in the strain rate, while ductile materials show elongation after yield. Furthermore, brittle materials often have higher-magnitude modulii of elasticity than do elastic materials: the slope of the σ-ε diagram is steeper for brittle materials.

The largest force that a material can sustain is called its maximum load (F_{max}), with accompanying maximum stress (σ_{max}). It should be noted that for ductile materials, maximum load might occur before failure of the material at ultimate load (F_{ult}).

Figure 4.1 illustrates σ-ε diagrams for generic brittle and ductile materials, and Figure 4.2 illustrates σ-ε diagrams for some types of stone.

---

[8] C.L. Searls, "Group Report: How Can We Diagnose the Condition of Stone Monuments and Arrive at Suitable Treatment Programs?" 205.

Typical Stress-Strain Diagrams for Generic Materials

Elastic, ductile material   Brittle material
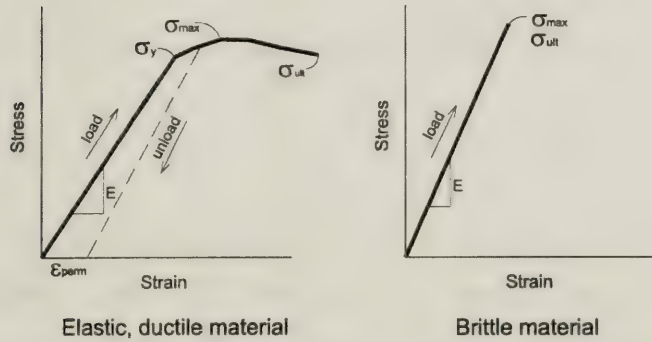
Figure 4.1

Stress-Strain Curves for Some Common Stones
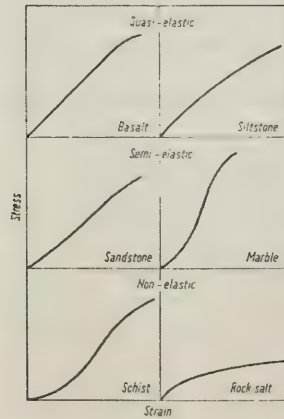
Figure 4.2

50

Bending stress (σ_b) is the result of applying a moment (M, a rotational couple) to an element or of applying a load eccentrically or perpendicularly to the member's longitudinal axis. The magnitude of bending stress depends on the cross-section of the element:

$$\sigma_b = \frac{My}{I},$$

where y is the distance from the centroid of the section to the point on the section where the stress value is desired, and I is the moment of inertia of the section (which describes the geometry of the section).[9] The resistance of a material to bending (flexure) is described by the modulus of rupture, found with the three-point bending test: a load is placed at the center of a beam supported at both ends. The load at failure is recorded and is used to calculate the moment applied to the section at failure. For a beam of rectangular cross-section, maximum bending stress occurs at the section edge ($y=d/2$, where d is the depth of the section), and the moment of inertia is $bd^3/12$ (b is the width of the section).

$$M_{max} = \frac{FL}{4} \quad \rightarrow \quad \sigma_{b,max} = \frac{FL}{4} \times \frac{d}{2} \times \frac{1}{\frac{1}{12}bd^3} \quad \rightarrow \quad \sigma_{b,max} = \frac{3FL}{2bd^2}$$

The modulus of rupture is defined as this maximum bending stress.

---

[9] $I = Ar_g^2$ where r_g is the radius of gyration of the section, defined as $\sqrt{\frac{I}{A}}$.

Mechanics of Composite Materials

Hosek and Panek report that analysis of stresses in a consolidated stone is based on the fact that the stone is a two-layer composite material and that the consolidant usually changes most mechanical properties of the stone, including modulus of elasticity and ultimate stresses.[10] Thus, if the contributions of a treated stone's layers to net reactions and deformations need to be determined, the composite stone must be separated into its constituents for analysis.

In the following discussion of composite mechanical action, it is assumed that particulate constituents are fully bonded to the matrix and that laminate layers are fully bonded to adjacent layers. When there is restraint such that the materials cannot act or react independently of one another, deformations or forces applied to one or both constituents of composite materials cause reactionary deformations or forces in the other constituents. This assumption presumes that the particles and matrix, or individual layers, react to each other when restrained by each other (an intact bond), by member end supports, or by volumetric confinement. The so-called "superposition method" aids in finding the reactions of the materials to applied forces. One of the reactions is designated as "redundant" and is considered as an unknown load; the resulting reactions are found by considering the deformations caused by given loads and the redundant reaction separately, but as functions of one another. These deformations are then combined –

---

[10] J. Hosek, and J. Panek, "Depth of Impregnation as the Criterium for Durability of Consolidated Stones," in *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix (Lausanne, Switzerland: Presse polytechniques romandes, 1985), 801.

superimposed – to obtain the final results after applying known boundary conditions to the system. Such boundary conditions can include a net zero deformation or displacement, or the condition that the reactionary forces of the constituent materials must sum to the applied force.[11] Figure 4.3 illustrates the type of composite mechanical action problem that will be considered in this thesis.

Restrained Composite Action of Column in Compression
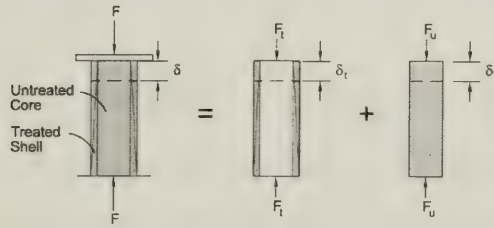
Vertical Sections

Figure 4.3

The following boundary conditions apply to this problem:

$$\delta = \delta_u = \delta_t \quad \text{and} \quad F = F_u + F_t.$$

These conditions allow the problem to be solved with the following equation sequence:

$$\delta_u = \frac{F_u L}{A_u E_u} \quad \text{and} \quad \delta_t = \frac{F_t L}{A_t E_t}$$

$$\text{so,} \quad \frac{F_t L}{A_t E_t} = \frac{F_u L}{A_u E_u}$$

---

[11] This method is given by Beer and Johnson, *Mechanics of Materials*, 60.

$$\text{and} \quad F_t = F - F_u$$

substituting, $\dfrac{F - F_u}{A_t E_t} = \dfrac{F_u}{A_u E_u} \quad \longrightarrow \quad \dfrac{F}{A_t E_t} - \dfrac{F_u}{A_t E_t} = \dfrac{F_u}{A_u E_u} \quad \longrightarrow \quad \dfrac{F}{A_t E_t} = \dfrac{F_u}{A_t E_t} + \dfrac{F_u}{A_u E_u}$

$$\frac{F(A_u E_u)}{A_t E_t + A_u E} = \frac{F_u(A_u E_u) + F_u(A_t E_t)}{A_t E_t + A_u E_u}$$

$$F A_u E_u = F_u(A_u E_u + A_t E_t)$$

$$F_u = \frac{F A_u E_u}{A_u E_u + A_t E_t}$$

and similarly, $F_t = \dfrac{F A_t E_t}{A_u E_u + A_t E_t}$.

Berthelot and Jones both suggest that the so-called "rule of mixtures" can be used to determine the apparent modulus of elasticity (E_{app}) of a composite material made of a dispersion of cube-shaped particles (p) in a matrix (m).[12] The rule of mixtures is expressed by the following equation:

$$E_{app} = E_m V_m + E_p V_p.$$

Jones extends this theory and applies it to the apparent forces and stresses of a composite material:[13]

$$\sigma_{app} = \sigma_m V_m + \sigma_p V_p.$$

It is unknown if experimental data published in the literature support this theory. One element of this thesis could be to prove or disprove this theory for the given composite material.

---

[12] Jones, *Mechanics of Composite Materials*, 159.
[13] Jones actually applies to fiber-matrix composites, but the extension to particle composites seems logical.

Knowing the forces in each constituent material allows the determination of stresses in each material as well as the deformation of the composite as a whole. If the cross-sectional areas and modulii of elasticity are not identical, the stresses in the materials differ such that a stress gradient is present in the composite as a whole. This is the very essence of composite action.

By considering only the modulus of elasticity of the materials, stiffer constituents resist deformation more easily, and stresses will concentrate in these materials. Because stress concentrates in them, stiffer materials may reach ultimate stress more quickly than less-stiff materials and may fail first, depending on maximum and ultimate stress.[14]

Structural Analysis and Design

Armed with the knowledge of pure and composite mechanical properties and basic mechanics equations, an engineer can analyze and design the material for predicted service loads the element must sustain without failure. It is essential to know if existing or future loads are applied axially (tension or compression), in shear, in bending, or in a combination of these modes. Only concentric axial loads will be considered in this thesis.

Columns are examples of structural elements that are loaded axially in tension or compression. The axial load is applied to the cross-section, which responds with deformation described by the equation

---

[14] Personal communication with Samuel Y. Harris.

$$\delta = \frac{kFL}{AE}.$$

L is the unsupported length of the column, but this is modified by the factor k, which accounts for the type of fixity at the column's ends. For rigidly supported (fixed) ends, k is 0.5; when the ends are simply supported (hinged), k is 1.0. The quantity kL is usually called the effective length (L_e) of the column.

For long columns in compression, there is a critical load (F_{cr}) at which the column will cease to be stable and will buckle. This critical load is defined by Euler's equation:

$$F_{cr} = \frac{\pi^2 EI}{L_e^2}.$$

By substituting Ar_g^2 for I and by substituting F_{cr} into the definition of stress, the critical stress (σ_{cr}) can be calculated with the following equation sequence:

$$\sigma_{cr} = \frac{F_{cr}}{A} \quad \rightarrow \quad \sigma_{cr} = \frac{\pi^2 EAr_g^2}{AL_e^2} \quad \rightarrow \quad \sigma_{cr} = \frac{\pi^2 E}{(L_e / r_g)^2}.$$

Thus, the critical stress of a column depends on the material of the column (its modulus of elasticity), the column's cross-section (the size and shape), and the structural configuration of the column (its effective length). Checking critical buckling stress of a long column is part of an engineer's checklist when analyzing columns, as will be done in an example in Chapter Five.

An engineer uses the basic stress equations along with the critical load and stress equations to design a new column. For example, if the project architect specifies a column to be constructed of a certain material, the ultimate compressive stress and

modulus of elasticity of that material will be known from testing or will be assumed from published values of a similar material. After applying a factor of safety so that the ultimate stress is theoretically never reached, the minimum cross-section (A_{min}) of the column can be determined. The engineer then crosschecks the selected material and section with the column's calculated critical stress. If the critical stress is less than the allowed ultimate stress, the engineer must use the critical stress as the limiting factor in the design and base A_{min} on it. In a similar manner, existing columns can be analyzed for existing stresses that are then crosschecked with the critical stress. If ultimate or critical stresses are approached or breached, the cross-section must be increased or the effective length must be decreased.

Material Failure

The ultimate goal of material characterization and testing, and of structural analysis and design, is to avoid material and structural failure. In ductile elastic materials, failure is often foreshadowed by large deformations or deflections; cracks form after yield stress is surpassed. Deformations may be elastic and objectively "safe" for the material; however, human occupants of buildings exhibiting perceivable deflection may deem the structure subjectively "unsafe" for occupation. In brittle materials, failure is announced by the initiation and propagation of cracks – often occurring simultaneously and swiftly. Cracks can be only aesthetically unpleasing, or they can warn of impending structural failure. In composite materials, cracks may not form in either constituent material; instead, shear stresses built-up at material interfaces may lead to debonding.

Crack Formation and Propagation

A specimen fractures when the stress acting on it, or around flaws or pre-existing cracks, reaches ultimate stress. Fracture can be described as a three-step process:[15]

- *Microcrack initiation* due to exceeding ultimate stress in localized areas, especially at material flaws or foreign inclusions.
- *Microcrack growth and propagation* in a stable fashion, often including crack intergrowth.
- *Microcrack growth and propagation* in an unstable fashion leading to wholesale failure of the material.

During the propagation stages, strain increases rapidly which leads to the growth of cracks.[16] The cracks may extend linearly in one plane, shear forward in the direction of the crack, or shear parallel and slip at angle to the crack's axis.[17] Water can effectively lubricate cracks to facilitate slip.[18] Stresses concentrate at the tip of the crack such that the material can see ultimate stress well below applied and apparent stress. Figure 4.4 illustrates crack initiation and growth as accompanied by increased loading of stone.

---

[15] Jones, *Mechanics of Composite Materials*, 339.
[16] Winkler, *Stone: Properties, Durability in Man's Environment*, 40.
[17] Jones, *Mechanics of Composite Materials*, 343.
[18] Walsh, "Deformation and Fracture of Rock," 97.
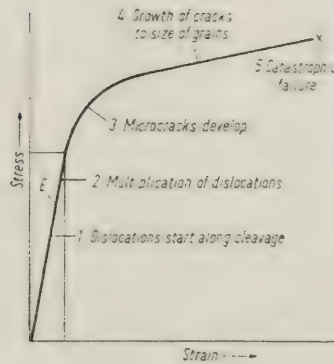
Figure 4.4

Reproduced from Winkler, *Stone: Properties, Durability in Man's Environment*, 39.

Figure 4.5 illustrates observable forms of fracture of cylinders (columns) at failure. The forms illustrate the manifestation of transverse (vertical) cracks parallel to the applied load and shear (diagonal) cracks. Walsh states that the type of fracture does not depend entirely on the material and that the configuration of the specimen is important. He notes that loading rate and temperature are also important – high rate and low temperature enhance brittleness. Deformation and fracture of rock cannot be characterized as either purely elastic or purely plastic, and fracture involves elements of both brittle and ductile behavior.[19]

---

[19] Walsh, "Deformation and Fracture of Rock," *Conservation of Historic Stone Buildings and Monuments* (Washington, DC: National Academy Press, 1982), 89 -90.
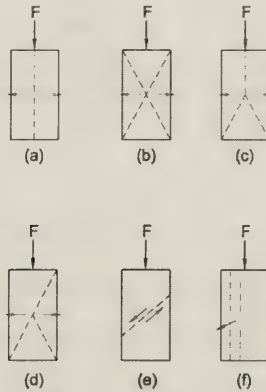
Figure 4.5

Failure types: (*a*) split, (*b*) cone, (*c*) cone and split, (*d*) cone and shear, (*e*) shear, (*f*) columnar.

Modified from *ASTM C39: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*; and Winkler, *Stone: Properties, Durability in Man's Environment*, Fig. 44.

Shear and Debonding

Laminate composites can exhibit through-body cracking, but more often cracks are confined to within one layer and stop at layer interfaces. More frequent than cracking, laminate composites display delamination – the separation of layers from one another due to shearing forces. The separation mechanism depends on the nature of the layers, the nature of the bonding material between them, the architecture of the layers, and the mode of mechanical loading.[20] Debonding may not mean that the layers adjacent to the failed bond can no longer carry load. After debonding each layer is independent and must rely

---

[20] Berthelot, *Composite Materials*, 236.

on its own (non-composite) properties for performance. Furthermore, any remaining bonded layers must carry higher load.[21] Jones carries out extensive study of laminar failure, including the phenomenon of free-edge delamination, in *Mechanics of Composite Materials*.

It should be noted that failure of a structural element might occur in an unexpected way: instead of crack formation or debonding, the element may fail by compressive buckling or by shearing at end supports. Thus, structural analysis and design uses proven design equations, but engineering judgment and common sense should also be used to predict probable failure mechanisms.

---

[21] Jones, *Mechanics of Composite Materials*, 259.

CHAPTER FIVE: MECHANICAL EFFECTS OF CONSOLIDATION

Overview

Consolidation of porous material is most often done in an attempt to stop "sugaring" of grains or loosening of flakes from the substrate's exposed face. The consolidation of the grain structure is the main goal of this treatment; it is not necessary in such applications to increase the load-bearing capacity of the deteriorated material.   In some cases, however, mechanical strengthening to increase load-bearing capacity of the deteriorated material is the key goal of consolidation, especially in fully-penetrating impregnation treatments. The application of the consolidant can eliminate the need for more drastic structural interventions such as removal of an "unsalvageable" structural element and its total replacement, or partial replacement of historic material with new stone, epoxy patches, or mortar infills.

Testing the mechanical properties of materials before and after chemical consolidation evaluates the treatment for its consolidating and strengthening effects.   Even when strengthening is not the main purpose of treatment, the mechanical properties of the material before and after treatment should be identified. Increases in compressive and/or tensile strength of the specimen may show efficacy of treatment, although some in the field argue against this idea.   Drastic increases in these parameters may alert to the potential of overstrengthening the substrate with treatment. As pointed out in Chapter Four, overstrengthening may lead to localized stress formation in the treated zone. Warke notes that if localized stress is applied to an area weakened by previous stress

events (which could include deterioration), further application of stress may trigger failure disproportionate to the magnitude of the stress applied.[1]

"Strengthening" includes increasing the tensile, compressive, and/or bending strengths of the substrate. Strengthening also often increases the in the modulus of elasticity (E) of the material, which can lead to embrittlement of the treated material. Embrittlement may lead to premature cracking under load, which can lead to water (or other deterioration agents) ingress, unappealing visual effects, and even nullification of the intended strength increase. Furthermore, a change in E changes the stress-strain behavior of the material, as will be illustrated later in this chapter.

In order to avoid overstrengthening, Sasse and Snethlage suggest that the treated material exhibit plastic behavior and a low E. They submit that overstrengthening a surface-treated zone is acceptable only if the following conditions are met:

- E of the consolidated zone is less than $1.5E$ of the sound material (the undeteriorated zone),
- the ratio E/σ_b of the treated zone does not exceed that ratio for the untreated zone,
- the strength of the treated zone decreases only behind the depth of the maximum of the mean moisture distribution curve,
- the penetration of the treatment must be deeper than the location of the maximum of the mean moisture distribution curve.

In addition, they suggest that a smooth slope of E over the treatment's penetration depth is more important than a large increase in strength. They also recommend that the biaxial

---

[1] P.A. Warke, "Inheritance Effects in Building Stone Decay," 35.

flexural strength of the treated stone be nearly equal that of the untreated stone. They suggest there be a homogeneous strength profile, with the strength of the treated zone increasing smoothly up to that of the untreated, undeteriorated zone. They clearly state, "Consolidation should return the strength of a stone maximum to its unweathered state."[2]

Literature Review

A literature review of previous researchers' work related to the mechanical properties of untreated and treated materials in conservation showed that most work has focused on natural stone. As such, the work on the mechanical properties of stone provides an important background for this thesis.

Sasse and Snethlage call for strength profiles of treated stone, like those illustrated in Chapter Three, to be constructed in order to closely assess strengthening as a function of depth of consolidant penetration. Koblischek constructed such a profile for untreated reed sandstone simultaneously with that of the stone treated with poly-silicic-acid-ethyl-ester.[3] His profiles show the tensile strength of the treated stone to be almost double that of the untreated stone to a depth of 1.2" (3 cm). The treatment penetrated the stone to a depth of 1.6" (4 cm), at which point the strength profile of the treated material merged with that of the untreated material. This example is the rare case: most researchers only

---

[2] Sasse, and Snethlage, "Methods for the Evaluation of Stone Conservation Treatments," 227 and 237. Interestingly, a 1996 article by these authors allows E of the treated zone to be $2.0E$ of the untreated zone.

[3] Peter J. Koblischek, "The Consolidation of Natural Stone with a Stone Strengthenr on the Basis of Poly-Silicic-Acid-Ethylester," in *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996* (Berlin: moller druck und verlag gmbh, 1996), 1188.

test (or publish) the global mechanical properties of the stone in their case studies without illustrating the strength as a function of depth. Constructing a strength profile can be a valuable and even crucial aspect of examining strength and/or modulus of elasticity increases; project time, budget and equipment may not always be available to the researchers for this type of painstaking analysis. Tables 5.1a (U.S. units) and 5.1b (metric units) offer a summary of selected published case studies and their mechanical testing results. It is quite apparent that not all studies test all the mechanical properties of stone. This could reflect the specific needs and goals of each study and/or the budget and equipment constraints of the project. It would be more valuable --especially to other researchers – if each case study determined and published all the mechanical properties of the untreated and treated stone.

Author, Date	Substrate	Treatment, Depth	Tensile Strength (psi)			Compressive Strength (psi)			Bending Strength (Mod. Of Rupture) (psi)			Modulus of Elasticity (ksi)		
			Before	After	% Change	Before	After	% Change	Before	After	% Change	Before	After	% Change
Clifton, 1984	Briar Hill sandstone 6" cubes	Epoxy, 0.35"	nr (*)	102	nr	8192	8802	7.4	nr	nr	nr	nr	nr	nr
		Silicone, nr	nr (*)	116	nr	8192	8454	3.2	nr	nr	nr	nr	nr	nr
		MMA, full	nr (*)	1710	nr	8192	8280	1.1	nr	nr	nr	nr	nr	nr
Clifton, 1984	Indiana limestone 6" cubes	Epoxy, nr	nr (*)	2640	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
		Silicone, nr	nr (*)	148	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
		MMA, nr	nr (*)	1150	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
Tabasso, et al., 1985	Lecce limestone 2" cubes	Ethyl silicate (OH), full	nr	nr	nr	2033	3014	48.3	nr	nr	nr	nr	nr	nr
		Acrylic resin (B-72), 0.2-0.4"	nr	nr	nr	2033	3228	58.8	nr	nr	nr	nr	nr	nr
		OH/B-72 mix, nr	nr	nr	nr	2033	3754	84.7	nr	nr	nr	nr	nr	nr
Zinsmeister, et al., 1988	Briar Hill sandstone size not given	Ethyl silicate (OH), 1"	nr	nr	nr	2431	4490	84.7	440	1062	141.4	nr	nr	nr
		Ethyl silicate (H), 1"	nr	nr	nr	2431	4972	104.5	440	951	116.1	nr	nr	nr
Saleh, et al., 1991	Egyptian sandstone 2" cubes (**)	Ethyl silicate (OH), nr	85	97	13.3	1422	3128	120.0	nr	nr	nr	nr	nr	nr
		Acrylic resin (B-72), nr	85	108	26.7	1422	3484	145.0	nr	nr	nr	nr	nr	nr
		MTMOS, nr	85	105	23.3	1422	2631	85.0	nr	nr	nr	nr	nr	nr
Wheeler, et al., 1992	Carrara marble 0.25" diameter, 3" length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	1624	2030	25.0	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	1624	1890	16.4	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	1624	1552	-4.4	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	1624	1610	-0.9	nr	nr	nr
Wheeler, et al., 1992	Wallace sandstone 0.25" diameter, 3" length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	1958	3785	93.3	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	1958	3553	81.5	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	1958	4161.5	112.5	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	1958	3625	85.1	nr	nr	nr
Wheeler, et al., 1992	Indiana limestone 0.25" diameter, 3" length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	1465	1972	34.6	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	1465	2161	47.5	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	1465	2697	84.1	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	1465	1697	15.8	nr	nr	nr
Tabasso, et al., 1994	Red tuff with black scoriae, 10.6" cubes	MMA, 2" (***)	nr	nr	nr	232	2712	1069.0	nr	nr	nr	nr	nr	nr
		PMMA, 2" (***)	nr	nr	nr	232	537	131.5	nr	nr	nr	nr	nr	nr
		Ethyl silicate, 2" (***)	nr	nr	nr	232	522	125.0	nr	nr	nr	nr	nr	nr
Rodrigues, et al., 1995	Granite 4.3" tall prisms	Ethyl silicate, full	nr	nr	nr	nr	nr	nr	nr	nr	nr	2248	2000	-11.0
		Epoxy, full	nr	nr	nr	nr	nr	nr	nr	nr	nr	2248	3103	38.0
Brus, et al., 1996	Horice sandstone 1.6" cubes	Ethyl silicate, full	nr	nr	nr	2677	3992	49.1	nr	nr	nr	nr	nr	nr
		Ethyl silicate/B-72 mix, full	nr	nr	nr	2677	4946	84.8	nr	nr	nr	nr	nr	nr
Christaras, 1996	Pentelic marble 1" diam., 0.4" high	not treated	1668	na	na	14428	na	na	nr	nr	nr	5568	na	na
Christaras, 1996	Yellow limestone 1" diam., 0.4" high	not treated	899	na	na	7062	na	na	nr	nr	nr	2248	na	na
Wendler, 1997	Yellow sandstone size not given	Ethyl silicate (OH), nr	nr	nr	nr	nr	nr	nr	1015	1595	57.1	E	approx 2E	100.0
		Elastified ethyl silicate, nr	nr	nr	nr	nr	nr	nr	1015	1668	64.3	E	approx 1.5E	50.0

Notes: (*) indicates the tension specimen were fabricated by crushing the stone and reconstituting it with consolidant.
    (**) indicates samples were taken from the monument – presumably "weathered" prior to testing
    (***) indicates strength measured for external consolidated shell
Unless noted, all values are assumed to be for dry, unweathered specimen

Table 5.1a

Abbreviations: nr = not reported, na = not applicable, MMA = methyl methacrylate, OH = Wacker OH,
H = Wacker H, MTMOS = methyl trimethoxysilane, PMMA = polymethyl-methacrylate

Mechanical Testing Results from Selected Case Studies Before and After Consolidation
(Metric Units)

Author, Date	Substrate	Treatment, Depth	Tensile Strength (MPa)			Compressive Strength MPai)			Bending Strength (Mod. Of Rupture) (MPa)			Modulus of Elasticity (GPa)		
			Before	After	% Change	Before	After	% Change	Before	After	% Change	Before	After	% Change
Clifton, 1984	Briar Hill sandstone 152 mm cubes	Epoxy, 9 mm	nr (*)	0.7	nr	56.5	60.7	7.4	nr	nr	nr	nr	nr	nr
		Silicone, nr	nr (*)	0.8	nr	56.5	58.3	3.2	nr	nr	nr	nr	nr	nr
		MMA, full	nr (*)	0.5	nr	56.5	57.1	1.1	nr	nr	nr	nr	nr	nr
Clifton, 1984	Indiana limestone 152 mm cubes	Epoxy, nr	nr (*)	18.2	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
		Silicone, nr	nr (*)	1.0	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
		MMA, nr	nr (*)	0.1	nr	nr	nr	nr	nr	nr	nr	nr	nr	nr
Tabasso, et al., 1985	Lecce limestone 5 cm cubes	Ethyl silicate (OH), full	nr	nr	nr	14.0	20.8	48.6	nr	nr	nr	nr	nr	nr
		Acrylic resin (B-72), 0.5 – 1.0 cm	nr	nr	nr	14.0	22.3	59.3	nr	nr	nr	nr	nr	nr
		OH/B-72 mix, nr	nr	nr	nr	14.0	25.9	85.0	nr	nr	nr	nr	nr	nr
Zinsmeister, et al., 1988	Briar Hill sandstone size not given	Ethyl silicate (OH), 2.5 cm	nr	nr	nr	16.8	31.0	84.5	3.0	7.3	143.3	nr	nr	nr
		Ethyl silicate (H), 2.5 cm	nr	nr	nr	16.8	34.3	104.2	3.0	6.5	116.7	nr	nr	nr
Saleh, et al., 1991	Egyptian sandstone 5 cm cubes (**)	Ethyl silicate (OH), nr	0.6	0.68	13.3	9.8	21.5	119.4	nr	nr	nr	nr	nr	nr
		Acrylic resin (B-72), nr	0.6	0.76	26.7	9.8	24.0	144.9	nr	nr	nr	nr	nr	nr
		MTMOS, nr	0.6	0.74	23.3	9.8	18.1	84.7	nr	nr	nr	nr	nr	nr
Wheeler, et al., 1992	Carrara marble 6.3 mm diameter, 75 mm length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	11.2	14.0	25.0	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	11.2	13.1	17.0	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	11.2	10.7	-4.5	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	11.2	11.1	-0.9	nr	nr	nr
Wheeler, et al, 1992	Wallace sandstone 6.3 mm diameter, 75 mm length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	13.5	26.1	93.3	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	13.5	24.5	81.5	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	13.5	28.7	112.6	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	13.5	25.0	85.2	nr	nr	nr
Wheeler, et al, 1992	Indiana limestone 6.3 mm diameter, 75 mm length	Ethyl silicate (OH), full	nr	nr	nr	nr	nr	nr	10.1	13.6	34.7	nr	nr	nr
		Ethyl silicate (H), full	nr	nr	nr	nr	nr	nr	10.1	14.9	47.5	nr	nr	nr
		Acrylic resin (B-72), full	nr	nr	nr	nr	nr	nr	10.1	18.6	84.2	nr	nr	nr
		MTMOS, full	nr	nr	nr	nr	nr	nr	10.1	11.7	1.8	nr	nr	nr
Tabasso, et al., 1994	Red tuff with black scoriae, 27 cm cubes	MMA, 5 cm (***)	nr	nr	nr	1.6	18.7	1068.8	nr	nr	nr	nr	nr	nr
		PMMA, 5 cm (***)	nr	nr	nr	1.6	3.7	131.3	nr	nr	nr	nr	nr	nr
		Ethyl silicate, 5 cm (***)	nr	nr	nr	1.6	3.6	125.0	nr	nr	nr	nr	nr	nr
Rodrigues, et al, 1995	Granite 11 cm tall prisms	Ethyl silicate, full	nr	nr	nr	nr	nr	nr	nr	nr	nr	15.5	13.8	-11.0
		Epoxy, full	nr	nr	nr	nr	nr	nr	nr	nr	nr	15.5	21.4	38.1
Brus, et al, 1996	Horice sandstone 4 cm cubes	Ethyl silicate, full	nr	nr	nr	18.46	27.53	49.1	nr	nr	nr	nr	nr	nr
		Ethyl silicate/B-72 mix, full	nr	nr	nr	18.46	34.11	84.8	nr	nr	nr	nr	nr	nr
Christaras, 1996	Pentelic marble 2.5 cm diam, 10 mm high	not treated	11.5	na	na	99.5	na	na	nr	nr	nr	38.5	na	na
Christaras, 1996	Yellow limestone 2.5 cm diam, 10 mm high	not treated	6.2	na	na	48.7	na	na	nr	nr	nr	15.5	na	na
Wendler, 1997	Yellow sandstone size not given	Ethyl silicate (OH), nr	nr	nr	nr	nr	nr	nr	7.0	11.0	57.1	1	approx 2/	100.0
		Elastified ethyl silicate, nr	nr	nr	nr	nr	nr	nr	7.0	11.5	64.3	1	approx 1.5/	50.0

Notes (*) indicates the tension specimen were fabricated by crushing the stone and reconstituting it with consolidant
(**) indicates samples were taken from the monument - presumably "weathered" prior to testing
(***) indicates strength measured for external consolidated shell
Unless noted, all values are assumed to be for dry, unweathered specimen

Table 5.1b

Abbreviations nr – not reported, na – not applicable, MMA – methyl methacrylate, OH – Wacker OH
H – Wacker H, MTMOS – methyl trimethoxysilane, PMMA – polymethyl-methacrylate

It is rare that treatment decreases the strength or modulus of elasticity of the subject stone: this case was only reported by Wheeler for bending strength when B-72 or methyl trimethoxysilane was applied to Carrara marble, and by Rodrigues and Costa for E when ethyl silicate was applied to granite. In all the other selected studies, consolidation increased strength and modulus of elasticity to some extent. Ethyl silicate increased compressive strength (σ_c) of various substrates between 48-125%. Acrylic resin increased σ_c between 59-145% in the selected cases. The greatest increase in σ_c was achieved with the application of methyl methacrylate to red tuff, as reported by Tabasso, et al.: the researchers recorded over 1000% increase in strength.

Tabasso, et al., 1985, and Tabasso, et al., 1994, measured compressive strength of the treated specimen after artificial/accelerated weathering (these values are not reported in Table 5.1). The compressive strength of the tuff samples (in the 1994 article) decreased after weathering, regardless of treatment type. The limestone samples (in the 1985 article) showed an interesting response to weathering: those treated with acrylic resin (B-72) and the OH/B-72 mix decreased in strength after weathering, while those treated with OH actually increased in strength after weathering. The increase in strength could be depend on the type of weathering used, but is probably an anomaly that should be disproved with further testing.

In the selected cases, most researchers did not measure modulus of elasticity. Rodrigues' and Costa's 1995 study specifically set out to examine contrasts in mechanical properties; however, they included elasticity information only for the granite specimen, but not for

the limestone specimen. The granite specimen experienced at 38% increase in E after treatment with epoxy.

Wendler's 1997 article was the only case study of those selected that published the stress-strain diagram from the mechanical testing. The diagram illustrates the biaxial flexural strength of sandstone and includes curves for the untreated stone, stone treated with ethyl silicate, and stone treated with elastified ethyl silicate. Although Wendler did not publish the actual values for E, the diagram allows readers to at least graphically infer the proportional values of E from one specimen type to the other two.

After doing their mechanical tests, none of the selected researchers used their data to quantitatively analyze the mechanics or structural performance of the observed increases in strength or modulus of elasticity; or, at least this type of analysis was not included in their articles as published. Most researchers acknowledge the fact that "overstrengthening is bad," but never *prove* it mathematically. Rodrigues and Costa (see above) visually examined the effects of consolidation on partially-impregnated samples by observing crack location and enhanced weathering as those phenomena related to penetration depth of the consolidant. From their findings, the authors warn of the potential danger of interfaces of mechanical properties between untreated and treated zones. They say this is a particular problem for "softer" stones, meaning porous limestones or tuffs. But again, these researchers did not analyze their data mathematically, or actually *use* the numerical measurements they recorded.

Visual observations may be more useful or practical in real life, especially since predicting mechanical properties or modeling the stone structure are often difficult tasks. It would be an interesting exercise, however, to at least use the strength measurements recorded so diligently by some researchers. In the following sections, the effects of deterioration and of subsequent consolidating strengthening will be examined using the basics of material mechanics and structural analysis introduced in Chapter Four.

Case Study: A Fictitious Pavilion

In the following case study, a fictitious open-air pavilion built in 1500 A.D. will be considered. The pavilion is assumed to be 50' by 50' (15.25 m by 15.25 m) in plan with a 20" (50.8 cm) diameter stone column at each corner. The columns are assumed to be monolithic (not drummed) and have smooth circular cross-sections (not fluted). Each column has an unsupported height of 30' (9.1 m), and the ends are assumed to be fixed (for the purposes of effective length). Figure 5.1 illustrates the pavilion.

Fictitious Pavilion for Case Study

Figure 5.1

70
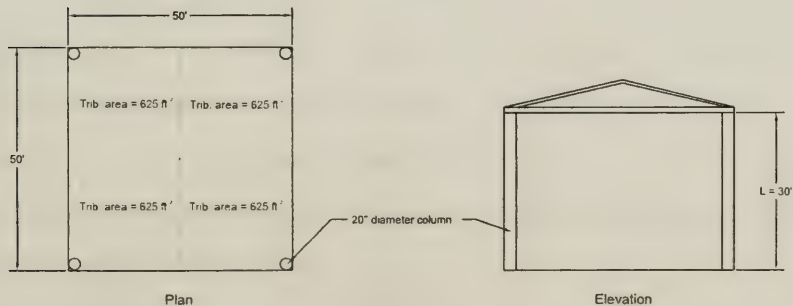
The loads are assumed to be (psf = pounds per square foot):

- *Dead load* (roof material, stone slab, ceiling beams): 100 psf
- *Roof live load*: 15 psf
- *Snow load*: 20 psf
- *Wind load*: 15 psf
- *Total load* = 150 psf

Each column carries one-quarter of the load applied to the roof so that the tributary area of each column is 25' by 25' or 625 ft². Thus, each column carries 625 ft² x 150 psf = 93,750 lb. This number is simply rounded to 100,000 lb.

Analysis of an Undeteriorated Column

The adequacy of the column as-built is analyzed first. It is common practice to apply a factor of safety to the design; for this analysis, a factor of safety of 1/2 will be applied to the actual maximum stress. This will be called the allowed stress (σ_{all}). If allowable stress is breached, the element is considered to fail.

$$\sigma_{applied} = \frac{F}{A} \rightarrow \sigma_{app} = \frac{100,000\,lb}{\pi(10")^2}$$

$$\sigma_{app} = 318\,psi$$

Table 2.1 reported that the compressive strength (σ_c) of marble ranges from 3000-15,000 psi, and E ranges from $0.3 \times 10^6 - 12 \times 10^6$ psi. $\sigma_c = 5000$ psi and $E = 6 \times 10^6$ psi will be assumed for this case. Thus, $\sigma_{all} = \frac{1}{2}\sigma_c = 2500$ psi.

$$\sigma_{app}\,(318\,psi) < \sigma_{all}\,(2500\,psi)$$

The marble pavilion column is adequate for the imposed load. The critical load (σ_{crit}) for column stability is calculated:

71

$$\sigma_{crit} = \frac{\pi^2 E}{(L_e/r_g)^2}$$

$$r_{g,circle} = \sqrt{\frac{I}{A}} = \sqrt{\frac{1/4\pi r^4}{\pi r^2}} = \sqrt{\frac{r^2}{4}} = \frac{r}{2}$$

$$\sigma_{crit} = \frac{\pi^2 E}{\dfrac{L_e^2}{r^2/4}} = \frac{\pi^2 r^2 E}{4 L_e^2}$$

$$L_e = 0.5\, L_{actual} = 0.5\,(30') = 15' = 180"$$

$$\sigma_{crit} = \frac{\pi^2 (10")^2 (6 \times 10^6\,\text{psi})}{4(180")^2} = 45,690\,\text{psi}$$

Because $\sigma_{crit} > \sigma_{all}$, σ_{all} controls the design. The column is adequate in stress and stability.

Analysis of a Deteriorated Column

Winkler reported a case in which 0.04-0.06" (1-1.57 mm) loss of surface of structural marble occurred over a fifty-year period by dissolution.[4] The fictitious pavilion is 500 years old; if Winkler's reported dissolution rate occurs on the pavilion's marble, and is attributed to pollution since the Industrial Revolution (beginning in 1800), a minimum loss of 0.16" of marble has occurred in the past 200 years. Assuming loss is total and the remaining stone is undeteriorated and not weakened, a core with radius of 9.84" remains. Figure 5.2 illustrates the loss.

---

[4] In W.D. Keller, "Progress and Problems in Rock Weathering Related to Stone Decay," in *Decay and Preservation of Stone, Engineering Geology Case Histories Number 11,* edited by Erhard M. Winkler (Boulder, CO: The Geological Society of America, 1978), 40.

Deteriorated Column

Undeteriorated Column — Deteriorated Column — Material loss
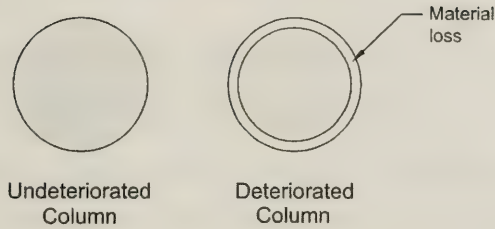
Figure 5.2

$$\sigma_{app,d} = \frac{100,000\,\text{lb}}{\pi[10 - 2(0.16")]^2}$$

$\sigma_{app,\,d} = 340\,\text{psi} < 2500\,\text{psi}.$

The stress increases 7% due to the reduction in cross-section, but the increased actual stress is still well below the allowed stress. In this case of slow loss of surface material, the choice to consolidate is made for aesthetics' sake, unless an increased rate of loss is anticipated.

What is the allowed reduction in cross-section before stress or stability failure ensues?

For strength: $\quad \sigma_{max} = \frac{F}{A_{min}} \rightarrow A_{min} = \frac{F}{\sigma_{all}}$

$$A_{min} = \frac{100,000\,\text{lb}}{2500\,\text{psi}} = 40\,\text{in}^2$$

$r_{mn} = 3.6"$ for strength

For stability: $\quad \sigma_{crit} = \frac{\pi^2 r^2 E}{4L_e^2} \rightarrow r^2{}_{crit} = \frac{\sigma_{crit}\,4L_e^2}{\pi^2 E}$

$$r_{crit} = \sqrt{\frac{\sigma_{crit} 4 L_e^2}{\pi^2 E}} = \sqrt{\frac{5000(4)(180")^2}{\pi^2 (6 \times 10^6 \text{ psi})}} = 3.3" \text{ for stability}$$

The column needs to have a minimum radius of 3.6" for strength; the column can lose a "shell" of material that is 6.4" thick (retaining 13% its original cross-sectional area). Loss of this magnitude could occur with spalling due to freeze-thaw cycling; loss of this magnitude due to dissolution is unlikely except in extreme environments.

Analysis of a Consolidated Column: Full Penetration of Section

In their 1985 article, Tabasso, et al., reported a 48.3% strength increase between untreated undeteriorated Lecce limestone and undeteriorated limestone treated with ethyl silicate consolidant. The consolidant had fully penetrated their 5 cm cubic specimens. If this treatment is applied to the fictitious column (now made of Lecce limestone) and fully penetrates the 20"-diameter cross-section, the allowable load on the column can increase. (See Table 5.1a: $\sigma_c = 2033$ psi.)

$$F_{all, \, before} = \sigma_{all, \, unstrengthened} \, A$$
$$F_{all, \, before} = (1017 \text{psi})(314 \text{in}^2) = 319,338 \text{ lb}$$

$$F_{all, after} = \sigma_{all, \, strengthened} \, A$$
$$F_{all, \, after} = (1507 \text{psi})(314 \text{in}^2) = 473,198 \text{ lb}$$

The additional allowed 150,000 lb could represent the weight of new roof-top mechanical equipment, new roofing layers, or the like. This model assumes that there is no partial deterioration on the column's surface, that the treated material is pure, and that its mechanical properties are homogeneous. The model assumes no composite action occurs.

74

If, as shown by Wendler (see above), this increase in strength is accompanied by a 100% increase in modulus of elasticity, the material becomes twice as stiff. In the case of full penetration, the theoretical structural behavior is not hindered; in fact, deformation under load will be reduced 50% and the critical stress for stability will be double that of the untreated specimen. The stiffer the material, however, the more vulnerable it is to cracking, as discussed previously.

Analysis of a Consolidated Column: Partial Penetration of Section

When the consolidant does not fully penetrate the section, a composite material is formed. Three cases will be considered: the consolidant penetrates the deteriorated material up to the undeteriorated core, the consolidant does not fully penetrate the undeteriorated section, and the consolidant fully penetrates the deteriorated zone and partially penetrates the undeteriorated section. In the following equations, the subscript *ud* refers to untreated deteriorated material, *uu* refers to untreated undeteriorated material, *td* refers to treated deteriorated material, *tu* refers to treated undeteriorated material. The subscript *app* refers to the applied force or stress.

Partial Penetration of Undeteriorated Material

Rodrigues' and Costa's 1995 granite study showed that with application of epoxy, *E* material increased from 2248 ksi (kilopounds per square inch) to 3103 ksi – a 38% increase. If the pavilion's columns are made of this granite and are treated such that the consolidant penetrates the 2" into the surface, a composite column results (Figure 5.3). In this model, it as assumed that the treated and untreated materials are fully bonded to

75

Partial Penetration of Undeteriorated Material
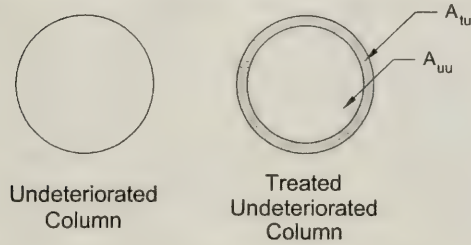
Figure 5.3

one another. One can assume an original strength of the granite of 14,000 psi (Table 2.1) and that the treated material experienced a 10% increase in strength (to 15,400 psi) in the penetration depth. The effect of strengthening and stiffening is examined below.

$$A_{tu} = \pi r_{total}^2 - \pi r_{uu}^2 = \pi(10")^2 - \pi(8")^2 = 113 \text{ in}^2$$

$$A_{uu} = \pi r_{uu}^2 = \pi(8")^2 = 201 \text{ in}^2$$

With the equation from Chapter Four, the resultant forces and stresses in the untreated undeteriorated material (F_{uu}) and treated undeteriorated material (F_{tu}) can be found.

$$F_{uu} = \frac{F_{app} A_{uu} E_{uu}}{A_{uu} E_{uu} + A_{tu} E_{tu}} = \frac{(100,000 \text{ lb})(201 \text{ in}^2)(2248 \times 10^3 \text{ psi})}{(201 \text{ in}^2)(2248 \times 10^3 \text{ psi}) + (113 \text{ in}^2)(3103 \times 10^3 \text{ psi})} = 56,306 \text{ lb}$$

$$F_{tu} = \frac{F_{app} A_{tu} E_{tu}}{A_{uu} E_{uu} + A_{tu} E_{tu}} = \frac{(100,000 \text{ lb})(113 \text{ in}^2)(3103 \times 10^3 \text{ psi})}{(201 \text{ in}^2)(2248 \times 10^3 \text{ psi}) + (113 \text{ in}^2)(3103 \times 10^3) \text{psi}} = 43,694 \text{ lb}$$

$$\sigma_{uu} = 280 \text{ psi} < 7000 \text{ psi allowed}$$

$$\sigma_{tu} = 387 \text{ psi} < 7700 \text{ psi allowed.}$$

The treated shell carries 38% more stress than the untreated core due to the increase in stiffness alone (the increase in strength only allows more load to be placed on the column). Because granite is so strong and stiff, there are no possible deleterious effects

76

when this composite column in compressed. If the stone was much weaker, stress due to loading the stiffer material could cause failure. Application of the strengthening treatment proves to be deleterious in this situation.

Examining Sasse's and Snethlage's allowed increase of E_t to $1.5E_u$ illustrates the case of stiffening. E_t for the granite will be $(1.5)(2248\text{x}10^3) = 3372\text{x}10^3 \text{psi}$.

$$F_{uu} = \frac{F_{app} A_{uu} E_{uu}}{A_{uu} E_{uu} + A_{tu} E_{tu}} = \frac{(100{,}000\,\text{lb})(201\,\text{in}^2)(2248\text{x}10^3\,\text{psi})}{(201\,\text{in}^2)(2248\text{x}10^3\,\text{psi}) + (113\,\text{in}^2)(3372\text{x}10^3\,\text{psi})} = 54{,}251\,\text{lb}$$

$$F_{tu} = \frac{F_{app} A_{tu} E_{tu}}{A_{uu} E_{uu} + A_{tu} E_{tu}} = \frac{(100{,}000\,\text{lb})(113\,\text{in}^2)(3372\text{x}10^3\,\text{psi})}{(201\,\text{in}^2)(2248\text{x}10^3\,\text{psi}) + (113\,\text{in}^2)(3372\text{x}10^3\,\text{psi})} = 45{,}749\,\text{lb}$$

$$\sigma_{uu} = 270 \text{ psi}$$

$$\sigma_{tu} = 404 \text{ psi}$$

The treated shell carries 33% more stress than the untreated core due to the increase in stiffness alone. Similarly, if E_t is $2.0E_u$, $\sigma_{uu} = 234$ psi and $\sigma_{tu} = 468$ psi – the treated shell carries twice the stress of the untreated core. Thus, applying consolidating treatments to undeteriorated stone may ward off future deterioration, but it also increases the stress and implies that the treated material will carry more load.

Impermeable films or pore-filling consolidants trap liquid water or water vapor behind them. The untreated material can then continue to deteriorate behind the treated shell – either via freeze-thaw heave or dissolution (or, in the case of granite, the feldspars could kaolinize and revert to clay). Thus, the treated shell may have to carry the entire load applied to the column:

77

$$\sigma_{opp} = \frac{100,000\,\text{lb}}{113\,\text{in}^2} = 885\,\text{psi}$$

885 psi < 7700 psi allowed, so the treated shell can hold the load alone. If the penetration depth was much less and the material much weaker, the shell could fail under the applied load. This failure is seen as spalling of the shell from the deteriorated core. Again in this situation, application of the strengthening treatment can be deleterious.

Incomplete Penetration of Deteriorated Material

The case of the consolidant not fully penetrating the deteriorated material will be considered next. As Figure 5.4 shows, the result is a composite of three materials: the treated deteriorated (*td*) material, the untreated deteriorated (*ud*) material, and the untreated undeteriorated (*uu*) material.

Incomplete Penetration of Deteriorated Material

Undeteriorated Column

Treated Deteriorated Column

A_{td}
A_{uu}
A_{ud}
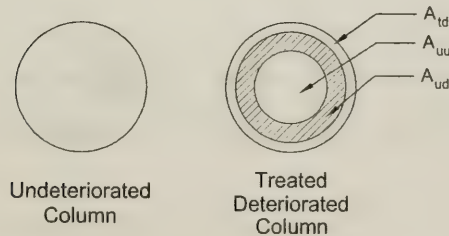
Figure 5.4

The boundary conditions for this case are:

$$\delta_{uu} = \delta_{ud} = \delta_{td} \quad \text{and} \quad F_{app} = F_{uu} + F_{ud} + F_{td}$$

$$\frac{F_{uu}L}{A_{uu}E_{uu}} = \frac{F_{ud}L}{A_{ud}E_{ud}} = \frac{F_{td}L}{A_{td}E_{td}}$$

These boundary conditions are insufficient for solving the problem; the problem remains indeterminate with three unknown forces (F_{uu}, F_{ud}, and F_{td}) but only two equations. Finite element analysis (either by hand or computer) of the situation could solve the problem, but this is beyond the scope of this thesis. In order to solve the problem, it will be assumed that the modulus of elasticity of the treated deteriorated zone (E_{td}) equals that of the untreated undeteriorated zone (E_{uu}), and that the area of the treated deteriorated zone (A_{td}) equals the area of the untreated undeteriorated zone (A_{uu}). This implies that $F_{td} = F_{uu}$. This is a preferred case; in fact, most conservators argue that the strength and modulus of elasticity of the treated material should equal that of the untreated undeteriorated material.

From the assumptions and boundary conditions, the resultant forces are found. Substituting $F_{td} = F_{uu}$ into the force condition: $F_{app} = 2F_{uu} + F_{ud} \rightarrow F_{ud} = F_{app} - 2F_{uu}$

The deformation relation becomes:
$$\frac{F_{uu}}{A_{uu}E_{uu}} = \frac{F_{app} - 2F_{uu}}{A_{ud}E_{ud}}$$

$$\frac{F_{uu}A_{ud}E_{ud}}{A_{uu}E_{uu} + A_{ud}E_{ud}} = \frac{F_{app}A_{uu}E_{uu} - 2F_{uu}A_{uu}E_{uu}}{A_{uu}E_{uu} + A_{ud}E_{ud}}$$

$$F_{uu}A_{ud}E_{ud} = F_{app}A_{uu}E_{uu} - 2F_{uu}A_{uu}E_{uu}$$

$$F_{uu}(A_{ud}E_{ud}+2A_{uu}E_{uu}) = F_{app}A_{uu}E_{uu}$$

$$F_{uu} = \frac{F_{app}A_{uu}E_{uu}}{A_{ud}E_{ud} + 2A_{uu}E_{uu}}$$

Material assumptions: Both the treated deteriorated and untreated undeteriorated limestone have σ_c = 2000 psi (Table 2.1) and E = 1.4x10^6 psi. The untreated deteriorated limestone has decreased strength – assume σ_c = 1000 psi and E = 0.7x10^6 psi. Assume heavy deterioration such that A_{uu} = 100 in^2 remains. $A_{ud} = A_{total} - A_{uu} - A_{td}$ = 114 in^2. The core of untreated undeteriorated material has a radius of 5.64". The band of untreated deteriorated material is 2.61" wide. This means that the depth of penetration of the treatment is 1.75" – a realistic value.

$$F_{uu} = \frac{(100,000\ \text{lb})(100\ \text{in}^2)(1.4\text{x}10^6\ \text{psi})}{(114\ \text{in}^2)(0.7\text{x}10^6\ \text{psi}) + 2(100\ \text{in}^2)(1.4\text{x}10^6\ \text{psi})} = 38,910\ \text{lb}$$

F_{td} = 38,910 lb and F_{ud} = 22180 lb.

$\sigma_{uu} = \sigma_{td}$ = 389.1 psi < 1000 psi allowed

σ_{ud} = 195 psi < 500 psi allowed

If the untreated deteriorated material eventually loses its strength capacity, the problem becomes interesting. The system then becomes a "column inside a shell": the shell of the treated deteriorated material is outside of and separated from the untreated undeteriorated core. These elements are not bonded together, so each acts independently of the other. This means the shell of treated material must carry the full applied load, as must the core of sound material.

$$\sigma_{td} = \sigma_{uu} = \frac{100,000\,\text{lb}}{100\,\text{in}^2} = 1000\,\text{psi} = 1000\,\text{psi allowed.}$$

This is a critical case: the two elements are on the verge of being overstressed. Thus, consolidation is only as good as its depth of penetration; if not all of the deteriorated material is consolidated, potential for failure of the structural member exists. Application of the consolidant can be deleterious in the future if it does not fully penetrate the deteriorated material.

Partial Penetration of Undeteriorated Core

Lastly, the case of the consolidant penetrating the deteriorated material fully plus partially penetrating the undeteriorated material will be examined (Figure 5.5). This is the type of penetration recommended almost universally by conservators. The model assumes that the treatment locks together the treated deteriorated shell and the undeteriorated core. Like the above case, the system is a composite of three materials: the treated deteriorated shell, a treated undeteriorated shell, and an untreated undeteriorated core. This model assumes the three components are fully bonded to each other and that consolidation strengthens the undeteriorated material beyond its original strength.

81

Partial Penetration of Undeteriorated Core

Undeteriorated
Column

Treated
Deteriorated
Column
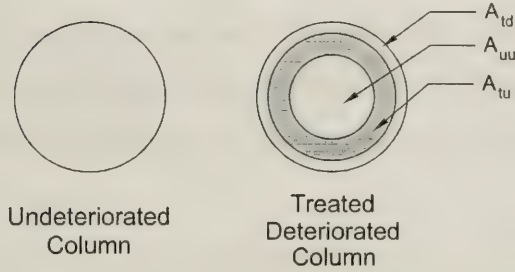
Figure 5.5

Material Assumptions: Both the treated deteriorated and untreated undeteriorated limestone have $\sigma_c = 2000$ psi (Table 2.1) and $E = 1.4 \times 10^6$ psi. The treated undeteriorated limestone has increased strength and stiffness – assume $\sigma_c = 2800$ psi and $E = 2.0 \times 10^6$ psi. As before, assume that $A_{uu} = A_{td} = 100$ in.^{2}. $A_{tu} = A_{total} - A_{uu} - A_{td} = 114$ in^2. The core of untreated undeteriorated material has a radius of 5.64". The band of treated undeteriorated material is 2.61" wide. This implies a penetration depth of 4.36". The equations for resultant forces are derived similarly as those for the "incomplete penetration" case above.

$$F_{uu} = \frac{F_{app} A_{uu} E_{uu}}{A_{tu} E_{tu} + 2 A_{uu} E_{uu}}$$

$$F_{uu} = \frac{(100{,}000 \text{ lb})(100 \text{ in}^2)(1.4 \times 10^6 \text{ psi})}{(114 \text{ in}^2)(2.0 \times 10^6 \text{ psi}) + 2(100 \text{ in}^2)(1.4 \times 10^6 \text{ psi})} = 27560 \text{ lb}$$

$$F_{td} = 27560 \text{ lb and } F_{tu} = 44880 \text{ lb}$$

$$\sigma_{uu} = \sigma_{td} = 276 \text{ psi} \quad \text{and} \quad \sigma_{tu} = 393 \text{ psi}$$

In this case, all elements of the composite material can sustain the applied load. If the untreated undeteriorated core degrades behind the treated shells (which are assumed to still be bonded to each other), the stress in the shells can be determined using the equations from the "partial penetration of undeteriorated material" case (that is, the case of a two-material composite).

$$F_{tu} = \frac{F_{app} A_{tu} E_{tu}}{A_{td} E_{td} + A_{tu} E_{tu}} = \frac{(100{,}000\,\text{lb})(114\,\text{in}^2)(2\text{x}10^6\,\text{psi})}{(100\,\text{in}^2)(1.4\text{x}10^6\,\text{psi}) + (114\,\text{in}^2)(2\text{x}10^6\,\text{psi})} = 61{,}956\,\text{lb}$$

and $F_{td} = 38044$ lb

$\sigma_{tu} = 543$ psi   and $\sigma_{td} = 380$ psi

The applied stress is higher in the undeteriorated shell element than in the three-material composite, but the stress is still below the material's allowed stress.

Loading and Stress Distribution

Two very important points should be made following the above examples. The first is that the case study assumes that the loads are applied such they are distributed uniformly to all portions of the cross-section; that is, no concentrated point loads were applied to the very center (the core) of the column. In design practice, point loads are distributed uniformly over supporting elements via bearing plates or, in the case of columns, via column capitals.

The second important point is that the case study assumes new loading of the treated material. In fact, if the consolidant is applied to an element already loaded and deformed (i.e., in an existing stress state), there is no change in stress state of the element.

83

Furthermore, if the consolidant is applied to a material already in a stressed state, there may not be a load or stress re-distribution. If application of the consolidant, however, triggers an increase in the material's volume, then application alone could cause redistribution of stresses with consequences similar to those described in the examples.

Load changes and stress redistributions are very likely events in the course of adaptation and preservation of an historic building. Removal or decrease in dead and/or live load occurs when a structural element is shored or supported during construction and treatment, or after a building is "mothballed" and becomes unoccupied. Reapplication or increase in dead and/or live load occurs after removal of shoring or reoccupation of the building. Changes in the type of load can also change the stress state of the element. If load was previously applied uniaxially and concentrically to a column element (as in the examples), the compressive stress is uniform across the cross-section of each untreated and treated zone and shearing stress does not develop. If the load is applied eccentrically, non-uniform stress distribution occurs not only across the entire section of the element, but also across each untreated/treated zone. Eccentrically-applied axial loads or lateral loads (wind, earthquake, etc.) cause bending and shearing stresses, which must be transferred across bonded planes of the different zones. It is in this case that shear and debonding failure can occur between the zones at the so-called shear plane.

Summary

Consolidants have been shown to effectively mitigate stone deterioration, and some even reinstate some or all of the substrate's mechanical strength. Because they may

84

"overstrengthen" or form pore-blocking crusts, application of consolidants can actually be detrimental to stone. Many researchers have quantified these phenomena, but few have actually explained why, how, or to what extent over-strengthening or crust formation can be detrimental to the structural performance of the stone. The case studies presented in this chapter quantitatively illustrate the stress relationships between treated and untreated materials.

The materials used in the case study were quite strong – it is unlikely that a structural column will be made of weak tuff stone, or the like. Thus, the case studies show that consolidation is usually not *structurally* necessary unless deterioration penetrates very deeply into the section. The case studies also show that as thin treated shells become stronger and stiffer, they take more of the applied load. This could lead to overstress if the stone is initially weak. The most critical case is that of material deterioration occurring behind a treated shell: if deteriorated material loses all of its bearing capacity, the remaining treated shell and untreated core can fail if applied load exceeds the thin or weakened materials' strengths.

CHAPTER SIX: TESTING PROGRAM

Objectives of the Testing Program

Compiling the preceding literature review made it apparent that more research is needed which considers the mechanical properties and composite action of consolidated material. For this thesis, a testing program was devised to measure the compressive strength and modulus of elasticity of a pure untreated material, a pure treated material, and a composite material with treated and untreated zones. In addition, failure mechanism, crack propagation, and possible shearing at the treated-untreated interface of the composite material during compression testing would be observed. For this study, concrete was the chosen substrate and ethyl silicate (Conservare OH) was the chosen treatment. Reasons for these choices will be discussed later in this chapter.

The objective of measuring compressive strength and modulus of elasticity is to examine the consolidant's effects on the load-bearing and stress properties of the substrate; examining the consolidant's efficacy in slowing or stopping deterioration was *not* an objective of this thesis program. The results of this testing program will provide actual measurements for analysis like that of Chapter Five. The testing program for this thesis is based in-part on that of J. Delgado Rodrigues and D. Costa from their 1995 study.[1] The present testing program was completed between October 1999 and February 2000.

---

[1] "Occurrence and Behavior of Interfaces in Consolidated Stones," in *Structural Studies of Historical Buildings IV - Volume 1,* edited by C.A. Brebbia and B. Leftheris (Boston: Computational Mechanics Publications, 1995), 245-252.

This testing program set out to show that consolidation not only changes the compressive strength and modulus of elasticity, but increases the magnitude of these properties. This is considered a "before and after study" by Reedy and Reedy.[2] It was acknowledged in the beginning stages of the program, however, that treatment of the specimen might show *no* change in the mechanical properties. Reedy and Reedy call this a "null hypothesis," which shows that treatment has no effect and is the same as "doing nothing."

This chapter describes the method by which the testing specimen were produced and treated. Chapter Seven describes the compression testing and its results.

Production of Specimen

When formulating a treatment for a specific building, conservators (or architects or engineers) test the treatment on the building material – either using samples taken directly from the building or using stone specimen identical or similar to the material used on the building. In non-case specific research done only to add to the body of scientific knowledge, researchers use material that is available to them or that is likely to be treated in actual cases. Thus, stone conservation research usually focuses on limestone, sandstone and marble, because these materials are frequently used in construction and often exhibit deterioration. In many cases, the researchers simulate deterioration and treatment by crushing stone into small pieces or powder, reconstituting

---

[2] *Principles of Experimental Design for Art Conservation Research* (Santa Monica, CA: The J. Paul Getty Trust, 1992).

the stone with consolidant into molded specimens, then testing the efficacy of the treatment on their fabricated specimen.

The testing program for this thesis had no specific building case study, nor did it aim to test the benefits of a treatment on any specific building material. For these reasons, it was not imperative to acquire a specific type of stone. Although results of testing a specific type of stone would add to the body of knowledge about that stone and its treatment, the results of the testing would still be limited to that specific stone type and treatment. Instead, this program aims to add to the general body of knowledge in the conservation field, and should serve to direct future research on specific types of stone and treatments.

For this reason, concrete was chosen as the substrate. Concrete is inexpensive to produce, can be formed into any size or shape, and is highly reproducible with given constituent materials and mix proportions. The selection of concrete and ethyl silicate was made in light of the fact that the manufacturer of the chosen consolidant claims that it can be applied to concrete. Although conservation treatments have not historically been applied to concrete and no published studies of applying ethyl silicate to concrete were found in a literature search, many concrete buildings are deteriorating such that conservation treatments (including ethyl silicate) *could* actually be applied to concrete in the near future. Thus, concrete serves as both a generic substrate for this testing program and as a specific substrate that could be the focus of future conservation work.

Overview of Concrete

Concrete is made of three basic ingredients: hydraulic cement (usually Portland cement), mineral aggregates, and water. Admixtures such as set retarders, set accelerators, plasticizers, and air-entrainers can also be included in concrete. Concrete is a popular building material in modern construction because of its excellent mechanical properties and its economy.[3]

Constituent Materials

Portland cement is produced by burning limestone or chalk (CaO) with clay or shale (sources of SiO_2, Al_2O_3, and Fe_2O_3). The clinker from this process yields four materials: tricalcium silicate ($3CaO \cdot SiO_2$, or C_3S), dicalcium silicate ($2CaO \cdot SiO_2$, or C_2S), tricalcium aluminate ($3CaO \cdot Al_2O_3$, or C_3A), and tetracalcium aluminoferrite ($4CaO \cdot Al_2O_3 \cdot Fe_2O_3$, or C_4AF). Minor constituents or impurities like magnesium or alkalis affect the quality of the cement. There are eight types of Portland cement manufactured today; each has specific properties directed at certain performance goals.

Portland cement reacts with water to produce a strong and insoluble solid. The calcium silicates give Portland cement its hydraulic character and influence the strength of the cement. When Portland cement is combined with water, exothermic hydration reactions occur. In the course of hydration, every cement grain breaks up into millions of particles

---

[3] General information about concrete comes from: Kenneth Derucher, George P. Korfiatis, and A. Samer Ezeldin, *Materials for Highway and Civil Engineers,* 3rd ed. (Englewood Cliffs, NJ: Prentice Hall, 1994); and Sandor Popovics, *Concrete Materials: Properties, Specifications and Testing,* 2nd ed. (Park Ridge, NJ: Noyes Publications, 1992).

in the water to form a poorly crystallized calcium silicate hydrate gel. The hardening of concrete is caused by the interlocking and recrystallization of the gel particles as water evaporates or becomes chemically bound in the constituents. The hardened cement paste contains a considerable amount of pores of different sizes: gel pores (15-30Å in diameter), and larger voids left by the evaporation of water or from induced air-entrainment. Incomplete hydration decreases the volume of gel pores and increases the volume of larger capillary pores.

Both fine and coarse aggregates are used in concrete. While cement imparts the most strength to concrete, aggregates are used to control shrinkage of the curing concrete and fill bulk volume for economy's sake. Fine aggregate is defined as that which passes a No. 4 (3/16", 4.75 mm) sieve. Coarse aggregate is that which is retained or is larger than 3/16" (4.75 mm). Natural sands and gravels are the most commonly used aggregates. The aggregates should be chemically inert; that is, no deleterious salts or alkalis should be part of the aggregates. The cement binds the aggregates together, which produces a composite material of aggregate particles and cement matrix. Grading the sizes of the aggregates is important, and using equidimensional particles is usually preferred over using flat or elongated pieces. Both grading and size determine packing of the particles in the matrix, as well as eventual strength and durability of the concrete mix.

Water used in concrete should be free of impurities like salts, alkalis, or acids. The general requirement of water used in concrete is that it be potable. The amount of water needed in a concrete mix is determined by the desired workability of the plastic concrete

and defined by the water/cement ratio (which also influences strength of the hardened cement).

General Practice in Making and Testing Concrete

Many textbooks and design guidelines have been published to aid in determining the proportions of cement, aggregate, and water to make concrete of intended strength. After the amounts of each constituent have been chosen, they are mixed together either by hand or with a mechanical mixer. The cement and aggregates are mixed first, and then the water is added. When placed in to its mold or form, the concrete is rodded or vibrated to ensure no large voids due to the placing procedure remain in the plastic mix.

Hardening concrete must be placed in an environment conducive to complete hydration of the cement. In a laboratory environment, ASTM specifies the curing room be 73±3°F (23±2°C).[4] They suggest the use of a moist room or placing the specimen in a tank of water. To provide a moist environment in the field, wet burlap or plastic sheets are usually placed over exposed concrete. The moist environment is maintained as long as practical in the field, or until testing the lab specimen. Passing moisture through hardening concrete brings fresh alkali (calcium hydroxide) formed by the hydration reactions to the surface, which allows carbonation to a greater depth. This increases early strength attained by the concrete.

---

[4] ASTM C 192/C 192M - 95: Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.

ASTM and ACI (American Concrete Institute) have written standards for testing both the constituents of concrete and the cured concrete mix. Strength tests are used most often to evaluate the properties of hardened concrete. Compression tests are also used as a quality-control measure on the construction crews batching and using concrete. ACI mandates that at least three test cylinders for each batch of concrete be made, and that the compressive strength be tested twenty-eight days after testing (or sooner/later, depending on the strength required by the designer).[5]

Sizes of the Concrete Test Specimen

Most stone specimen used in conservation research and testing are small. The most popular specimen size is the 2" (5 cm) cube. Prisms have been used in many research projects: 2"x2"x3.9" (5 cm x 5 cm x 10 cm) is a frequently used specimen size. Some use cores: Wheeler, et al., used 0.25" (0.64 cm) diameter by 2.4" (6.0 cm) long marble core samples.[6] Testing done by ProSoCo, Inc. (who produces Conservare OH) specifies that 2" (5cm) diameter by 8" (20.3 cm) long cylinders of stone be sent to them for testing.[7] Larger specimen are infrequently used; however, Tabasso, et al., used 10.6" (27cm) cubes because they better reproduced the conditions encountered when treating

---

[5] *Building Code Requirements for Structural Concrete and Commentary (ACI 318-95)* (Farmington Hills, MI: American Concrete Institute, 1995).

[6] "Evaluation of Some Current Treatments for Marble," in *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 2nd International Symposium, Geneve, 19-21 November,* edited by D. Decrouvez, J. Chamay and F. Zezza (Geneva: Museum of Art and History, 1992(, 439-443.

[7] ProSoCo, Inc., "Test Program for Stone Consolidation (Preservation)," *ProSoCo Technical Bulletin 483-2* (Kansas City, KS: ProSoCo, Inc., n.d).

monuments.[8] Specimen size, then, is determined by availability of material, laboratory storage space, available equipment, objectives of testing, and the desire to reproduce actual field conditions.

For the current testing program, both 2" (5 cm) concrete cubes and 6" (15.25 cm) diameter by 12" (30.5 cm) long concrete cylinders were made. 2" cubes and 6" diameter cylinders are standard specimen sizes for ASTM concrete tests. A 2" cube was deemed small enough to ensure full penetration of the consolidant. The 6" cylinders were deemed large enough to more accurately portray failure mechanism, crack propagation, and composite/interface action than the 2" cubes. As a side note, it would have been difficult and expensive to obtain large specimen of building stone, and larger specimen would be more prone to geologic heterogeneity.

Characterization of the Concrete Constituents

The author purchased all the concrete materials for this thesis from George F. Kempf Building Material Supply Company in Philadelphia, Pennsylvania. Prior to designing the concrete mix used in this study, the material properties of the coarse aggregate, fine aggregate and Portland cement were determined.

---

[8] "Interaction Between Volcanic Tuff and Products Used for Consolidation and Waterproofing Treatment," in *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990,* edited by A. Elena Charola (Rome: ICCROM, 1994), 173-189.

Coarse Aggregate

The supplied coarse aggregate was crushed stone. It originated from several quarries; unfortunately, the customer representative from Kempf could not define the source quarries of the coarse aggregate. The author did not complete a petrographic examination of the coarse aggregate for this thesis. Visual description of the aggregate is in Appendix A. Appendix A also describes the procedures used and the raw data collected during testing coarse aggregate. Table 6.1 summarizes the findings of the sieve analysis for the coarse aggregate. Table 6.2 summarizes other physical properties of the coarse aggregate.

Sieve Analysis of Coarse Aggregate

Sieve and Mesh Opening	% Retained
No. 4 (4.7 mm)	85.3
No. 8 (2.38 mm)	13.9
No. 16 (1.18 mm)	0.5
No. 30 (0.6 mm)	0.0
No. 50 (0.3 mm)	0.0
No. 100 (0.15 mm)	0.0
No. 200 (0.075 mm)	0.0

Table 6.1

Physical Properties of Coarse Aggregate

Property	Value
Bulk specific gravity, dry	2.58
Bulk specific gravity, saturated surface dry	2.62
Solid unit weight	161.0 lb/ft^3
Dry-rodded unit weight	107.6 lb/ft^3
Moisture content	0.0%
Absorption	1.4%

Table 6.2

Fine Aggregate

The supplied fine aggregate is called "concrete sand" and was purchased by Kempf from the Better Materials Corporation. The author did not complete a petrographic examination of the fine aggregate for this thesis. Visual description of the fine aggregate is included in Appendix A. Better Materials Corporation (BMC) supplied their sieve analysis of the sand, and the author completed an independent sieve analysis. Appendix A includes the raw data from the author's sieve analysis. Table 6.3 summarizes the sieve analysis results.

Discrepancies between the values determined by the Better Materials Corporation and the author cannot be explained; for the purpose of this thesis, the values determined by the author were used in designing the concrete mix. For batching the concrete, all fine aggregate used in the concrete mix passed the No. 4 sieve.

Sieve Analysis of Fine Aggregate

Sieve and Mesh Opening	% Retained (BMC)	% Retained (author)
No. 4 (4.7 mm)	4	3.8
No. 8 (2.38 mm)	10	16.3
No. 16 (1.18 mm)	19	29.3
No. 30 (0.6 mm)	25	37.1
No. 50 (0.3 mm)	21	12.4
No. 100 (0.15 mm)	18	12.4
No. 200 (0.075 mm)	2	0.9
Fineness modulus	3.85	2.59

Table 6.3

Appendix A includes the raw data collected in determining other physical properties of the fine aggregate. Table 6.4 summarizes these properties.

Physical Properties of Fine Aggregate

Property	Value
Bulk specific gravity, dry	2.03
Bulk specific gravity, saturated surface dry	2.18
Solid unit weight	126.7 lb/ft^3
Moisture content	4.2%
Absorption	7.6%

Table 6.4

Portland Cement

The cement supplied was Allentown Type I Portland cement and was purchased by Kempf from the Allentown Cement Company, Inc. The chemical components of this material are listed in the material safety data sheet and are summarized in Table 6.5. As provided by Derucher et al.,[9] the bulk specific gravity of Portland cement is 3.14 and the unit weight is 195.9 lb/ft^3.

Chemical Components of Allentown Type I Portland Cement

Component	%
Tri-calcium silicate	20-70
Di-calcium silicate	10-60
Tetra-calcium-alumino-ferrate	5-15
Calcium Sulfate	2-10
Tri-calcium aluminate	1-15
Magnesium oxide	0-4
Calcium oxide	0-0.2
Crystalline silica	0-0.2
Chromates	1-0.005

Table 6.5

---

[9] *Materials for Civil & Highway Engineers,* 3rd Edition, 148.

<u>Water</u>

Potable tap water was used in mixing the concrete for the test specimens. ASTM C192-95 ("Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory") and ASTM C31-96 ("Standard Practice for Making and Curing Concrete Test Specimens in the Field") do not specify tests to analyze the water used. No admixtures were added to the concrete used in this thesis.

Concrete Mix Design

In designing a concrete mix, three principle factors are of importance: quality, workability, and economy of the concrete. Quality is measured by the strength and durability of the concrete. Workability describes the ease with which the concrete can be mixed and placed into its form without segregation of the aggregates from the cement paste. Economy takes into account effective use of materials, effective operation and ease of handling.[10]

For designing the concrete used in this thesis, the author began by using the "ACI Method" as outlined in Derucher, et al.[11] A trial batch used mix proportions based on that design guide. The water-cement ratio for the trial mix was 0.95, which is relatively high. The trial batch was indeed found to be "soupy," so the author adjusted the mix for Batch A to the proportions reported in Table 6.6. Although the design calculations were

---

[10] Derucher, et al. *Materials for Civil & Highway Engineers*, 136-137.
[11] Ibid., 142-148. These pages include tables used in the design equations as well as a step-by-step example concrete design problem.

for one cubic yard (1 yd^3, 0.76 m^3), the actual batch volume (and corresponding material weight) was based on both the capacity of the mixing pan and the number of cubes and cylinders to be produced from one batch. All constituents were weighed using U.S. units, and are reported as such.

Concrete Mix for Batch A

Component	Weight Needed for Batch Size
Portland cement	27.0 lb
Coarse aggregate	90.5 lb
Fine aggregate	50.0 lb
Water	12.75 lb

Table 6.6

This mix proved to be more workable with a water-cement ratio of 0.47. This mix yielded fifteen cubes and five complete cylinders – a practical amount to mix by hand using available equipment. Batch A specimen were used for preliminary treatment and testing.

Batches B – G for final treatment and testing used the mix described in Table 6.7. The amount of water was increased slightly to provide more workability and lower compressive strength (w/c = 0.51).

Concrete Mix for Batches B-G

Component	Weight
Portland cement	27.0 lb
Coarse aggregate	90.5 lb
Fine aggregate	50.0 lb
Water	13.75 lb

Table 6.7

As specified by ASTM C192-95, all molds for concrete should be made of a non-absorbent material (steel, cast iron, or other non-absorbent material).

ASTM C109-98 ("Standard Test Method for Compressive Strength of Hydraulic Cement Mortars Using 2" Cube Specimens") provides a standard for 2" cube molds. The authored designed and had fabricated stainless steel cube molds to conform to this standard. Fillipi Brothers of Philadelphia, Pennsylvania, fabricated the molds. Although Fillipi could not ensure the tolerances mandated by Table 1 in ASTM C109, the molds are of adequate quality for this research project. Five molds, each containing three cube compartments, were used. Figure 6.1 shows one of the cube molds.

Cube Mold

Figure 6.1

ASTM C470-94 ("Standard Specification for Molds for Forming Concrete Test Cylinders Vertically") mandates that cylindrical molds have a nominal height equal to twice the nominal inside diameter. Single-use molds may be made of sheet metal, plastic, or suitably treated paper products. The molds must not leak when filled with water or concrete. American Paper Products of Alpha, New Jersey supplied the cylinder molds used in the preparation of specimens for this thesis. These molds are single-use, paraffin-coated paper molds with metal bases. The nominal inside diameter is 6" and the nominal height is 12". The manufacturer states that the molds meet ASTM C470 requirements. Figure 6.2 shows one of the cylinder molds.

Cylinder Mold

Figure 6.2

100

Specimen Preparation: Mixing the Concrete

ASTM C192-95 provides the standard for making and curing concrete test specimens in the laboratory. The author batched all concrete specimens in the Systems Engineering Materials Lab in the Towne Building on the University of Pennsylvania campus. The materials were batched at ambient conditions of this lab: 79±5°F (26.1°C) and 33±5% relative humidity. Prior to batching, the materials were stored in thick paper bags provided by the supplier. The aggregates were not kept in a saturated or saturated surface dry condition.

Materials were weighed and hand mixed according to the procedure given by ASTM C192: the fine aggregate and Portland cement were mixed together until thoroughly blended, the coarse aggregate was thoroughly incorporated into the sand-cement mix, then the water was added. The ingredients were mixed in a metal pan using a metal hoe. The concrete was mixed until a homogeneous and workable consistency was achieved.

ASTM C109-98 specifies the method for placing 2" cubic specimens. No form release agent was applied to the stainless steel molds prior to placing the concrete. The concrete was placed in two lifts, and each lift was tamped thirty-two times with a rectangular tamper in the manner described by the specification. The top of the cube was struck off using a slicing motion with a metal trowel. A "tent" of plastic wrap was placed over the finished cube molds (Figure 6.3) and the concrete cured in the lab. The concrete was removed from the molds approximately twenty-four hours after placement. The cubes were then placed in a sealed plastic container and allowed to cure in the lab. After

Figure 6.3

approximately five days of curing, the rough edges of the cubes were ground smooth using a polishing wheel, tap water, and 80 grit silicon carbide abrasive. After polishing, the grit was rinsed from the surface with tap water.

ASTM C192-95 ("Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory") was the standard used in forming the concrete cylinders. The concrete was placed into each cylinder mold with a trowel in three equal lifts. Each lift was rodded twenty-five times with a metal tamping rod. The surface was struck off using a

Figure 6.4

slicing motion with a metal trowel. The top of each cylinder was covered with the plastic hood (Figure 6.4) provided by Alpha Paper Products. The hood was secured with masking tape to ensure a tight fit. The cylinders were cured in the lab. A moist room was not available; in anticipation of applying the treatment to the specimen, the author deemed it inappropriate to cure the concrete under water. The concrete was removed from the cylinder molds approximately twenty-four hours after placement. The cylinders were then wrapped in polyethylene trash bags and allowed to cure in ambient lab conditions until treatment. After approximately five days of curing, the cylinders were capped with Hi-Cap Capping Compound (a sulfur compound). After capping, the cylinders cured in the lab without the plastic wrap.

Visual Characterization of the Hardened Concrete

Figure 6.5 shows a cross-section of a hardened 2" concrete cube prior to treatment. The aggregate appears to be uniformly distributed. Voids with maximum size of 1/16" (1.5 mm) are scattered throughout the cross-section. These voids are actually desirable for treatment purposes. Appendix B includes an inventory of all cube and cylinder specimen, noting batch date, treatment date, and specimen testing use.

Cross-Section of Hardened Concrete

Fb15a - Untreated
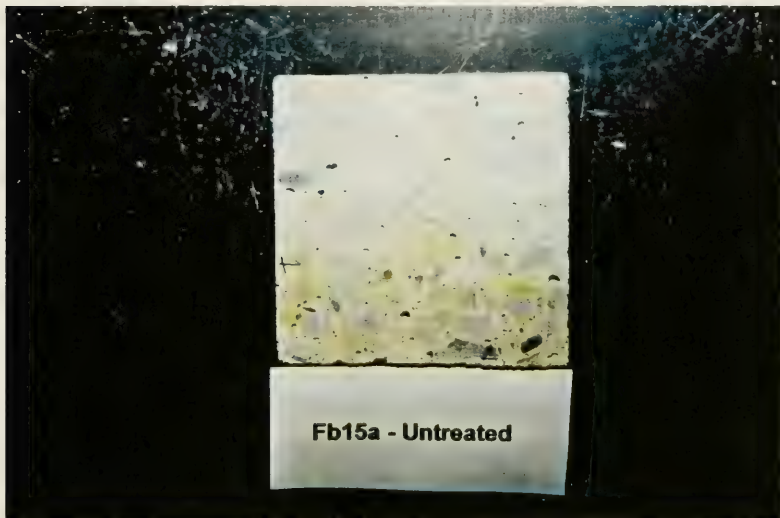
Figure 6.5

Treatment of Specimen

In choosing a suitable consolidant for the concrete specimen, the following criteria were used:

- appropriateness for application to concrete
- low viscosity for sufficient penetration into concrete
- practicality for application in the lab (this project) and in the field (actual use)
- availability of the product for purchase
- relative safety to environment and people.

Tiano, et al., completed a survey of products frequently used for consolidation in Italy. They found that Paraloid B-72, Rhodorisil 11309, Wacker OH, and Akeogard CO were among the products used, and Wacker OH was the most frequently used.[12] Wacker OH is an ethyl silicate – an alkoxysilane (tetraethoxysilane) – which has proven to be quite effective as a consolidant. Many researchers have found it to deeply penetrate stone substrates. As reported in Table 5.1, it can also increase the compressive strength of stone. Weber and Zinsmeister note, "No other types of consolidants have been studied so extensively and no other systems have ever had a longer track record of successful treatments than tetraethoxysilane-based products."[13] Wacker Chemie GmbH manufactures Wacker OH in Europe; ProSoCo, Inc., markets the same product in the United States but calls it Conservare OH. "OH" in the product name is an abbreviation for *ohne hydrophobierung* (German for "non-water repellent"), and should not be confused with the hydroxyl molecule O-H.

---

[12] P. Tiano, C. Filareto, A. Granato, and F. Piacenti, "Methods and Materials Used for the Conservation of Monumental Works in Italy," in *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer (Berlin: moller druck und verlag gmbh, 1996), 885-894.

[13] *Conservation of Natural Stone: Guidelines to Consolidation, Restoration and Preservation* (Ehningen, Germany: expert verlag, 1991), 63.

Overview of Tetraethoxysilane

Tetraethoxysilane is based on orthosilicic acid, which is a silicon atom surrounded by hydroxyl molecules. When alkyl groups (methyl, ethyl, etc.) replace the hydrogens, the compound is called silicate ester.[14] Silicate ester is defined as $Si(OR)_4$, where R is an alkyl group. Silicate ester hydrolyzes in the presence of water; hydrolysis is aided in some proprietary products by addition of a catalyst. During hydrolysis one alcohol molecule is formed and evaporates, and an acidic residue remains attached to the silicon atom. This molecule is called a silanol. When two acidic groups belonging to different silicic acid molecules interact, condensation (polymerization) takes place: one water molecule is released and a strong Si-O-Si bond is formed. Hydrolysis and condensation are combined in the chemical equation

$$Si(OR)_4 + 4H_2O \quad \rightarrow \quad SiO_2{\cdot}aq + 4ROH + 2H_2O.$$

The connection of silicon during condensation forms a three-dimensional network of amorphous hydrated silica.

The viscosity of commercial products containing liquid silicate ester is lower than that of water; with this, and the fact that hydrolysis occurs slowly, the chemical can penetrate deeply into stone before the silica gel binder ($SiO_2{\cdot}aq$) is formed. The alcohol by-product is not deleterious to stone. During the hardening (loss of structural water),

---

[14] Information about tetraethoxysilane is from: Charola, "Brief Introduction to Silanes, Siloxanes, Silicones and Silicate Esters," in *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration* (Paris: UNESCO, 1988) 313-314; Weber, "Stone Conservation - Planning and Execution," 15-22.

cracking of the deposited silica gel film can occur. Wendler notes, however, that in sandstones, there is little loss of durability of the consolidant.[15]

Lewin explains that application of silicon esters to stone is based on the reactivity of the OR groups with hydroxyl groups. If the hydroxyl group comes from the mineral structure of the stone, the silicon ester can react with it and bond to the surface of the mineral. After condensation, one end of the Si-O-Si backbone can bond to one grain and the other end can bond to an adjacent grain, effectively linking the grains. If the stone does not have hydroxyl groups, the consolidant may not link the minerals but instead may only fill the substrate's pores with silicate.[16]

The silicate acid ester-concrete interaction effects are unknown. Concrete and silicate acid ester are "like" materials in that they are both based on silicates. Lewin and Wheeler report that in the presence of alkalis, the liquid tetraethoxysilane reacts with water to set as a gel in less than one minute.[17] In tuff, Lukaszewics hypothesizes that silicate acid ester may crosslink the stone's aluminosilicate layers due to the reaction of their hydroxyl groups with those of the partly hydrolyzed alkoxysiloxanes.[18] Although the alkalis in concrete could be of great importance in considering penetration and gelling of the consolidant, the effects have not been further pursued for this thesis. The chemical

---

[15] "Materials and Approaches for the Conservation of Stone," 189.
[16] "The Current State of the Art in the Use of Synthetic Materials For Stone Conservation: Inorganic and Metal-Organic Compounds," 296-297.
[17] "Alkoxysilane Chemistry and Stone Conservation," in *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 831-844 (Lausanne, Switzerland: Presse polytechniques romandes, 1985), 833.
[18] "Application of Silicone Products in the Conservation of Volcanic Tuffs," 192.

bonding of the silicon backbone to the cement paste in concrete was not explored in this thesis; however, if the aggregate in concrete contains hydroxyl groups, mineral bridges may be formed from the aggregate to the silicate gel in the cement. For the purpose of this thesis, the penetration and consolidation mechanisms of ethyl silicate into concrete were not studied further; any strengthening of the treated concrete found in this study could be due to bridging and/or pore filling mechanisms.

Characterization of Conservare OH

Conservare OH (henceforth simply called OH) is a proprietary ethyl silicate product. It contains both diluting/carrying solvents and a catalyst. The material safety data sheet for OH lists 2-propanone, ethyl alcohol, 2-butanone, tetraethylorthosilicate, and an unnamed organic tin compound (the catalyst) as its chemical constituents. Table 6.8 summarizes other technical data supplied by ProSoCo.

Technical Data for Conservare OH

Property	Value
Active substance	Silicic ethyl ester
Specific gravity	0.938
Solids content (by weight)	75%
Solvent content (by weight)	25%
Flash point	36°F (2°C)
Weight	7.8 lb/gallon (0.92 kg/liter)

Table 6.8

Application Techniques for Consolidants: Literature Review

There are several ways of applying liquid consolidant to its substrate. The methods of application can be classified as long-time or short-time. Long-time techniques include

those that allow the consolidant to dwell on the surface of the substrate for an extended period of time (hours or days). Short-time techniques involve quick applications but usually mandate more than one application of consolidant be made. Below is a summary of specific techniques of long-time and short-time applications.

Long-time application
- full immersion (usually in closed container)
- partial immersion
- capillary absorption
- flowing stream of fluid over surface
- "pocket" method (pool of fluid is kept against vertical surface of substrate)
- poulticing
- continuous spray
- drip application with pipettes
- vacuum impregnation
- pressure impregnation

Short-time application
- Brushing in repeated cycles
- Low-pressure spraying in repeated cycles

Some suggest that full immersion may actually inhibit penetration of the consolidant into the specimen: over time the liquid gels and solidifies inside the stone and inhibits further penetration of consolidant, producing a zone of heavily consolidated material near the surfaces. Partial immersion is a popular method and can result in full penetration of small specimen. In this technique, the sample rests in a pool of consolidant, and the consolidant rises into the sample via capillarity. In some cases, the surfaces of stone not in contact with the product are wrapped in foil, tape, or wax to slow solvent evaporation.

Some conservators advocate pretreatment of the stone with the consolidant's carrier solvent or other wetting agents to enhance penetration.[19]

When doing laboratory trials to determine consolidant efficacy on a specific building, it is important to replicate as closely as possible the technique that will be used in actual field application. While full or partial immersion, vacuum, or pressure techniques may be practical for small objects, buildings are usually treated via brush or spray application. These techniques, however, may yield limited penetration depth if the substrate has low permeability.

To aid in developing an application technique for this thesis, a matrix listing application technique, curing conditions, and achieved penetration depth from previous researchers' work was developed. Table 6.9 reports a selection of case studies from this literature review.

Considering the expected low porosity and permeability of the concrete, it was decided to treat the 2" cubes via full immersion for several days. To simulate a field situation, the 6" cylinders were treated via brush application in several cycles.

---

[19] Anne Moncrieff, "The Treatment of Deterorating Stone with Silicone Resins: Interim Report," *Studies in Conservation* 21 (1976): 179-191.

Author, Date	Substrate, Size	Consolidant	Application Technique	Curing Time and Conditions	Penetration Depth	Notes
ProSoCo, 1996	any	Conservare OH	brush, spray or immersion (see notes)	flush surface with methyl ethyl ketone, cure 2-3 weeks at 50-90°F (10-32°C)	dependent on substrate	apply in three cycles: each cycle is three successive applications at 5-10 minute intervals; allow 20-60 minutes between each cycle
Boos, et al., 1996	German sandstone	modified elastic silicic-acid ester	full immersion (set in glass dish on glass beads, 2 minute soak 3 times with 30 seconds between immersions)	3 weeks	0.4" (10mm)	
Littman, et al., 1993	various sandstones, 2"x2"x4" (5cm x 5cmx10cm)	polyurethane	full immersion for 4 hours	28 days	(not noted)	
Useche, 1994	tuff, 2.2x2.2x3.4	various, including Wacker OH	full immersion 1-5 days	(not noted)	(not noted)	those specimen immersed for 1 day gave best compressive strength results
Weber, 1987	conglomerate stone, size not reported	Wacker OH	full immersion: three immersions of 2 minutes each	3 weeks	>20mm	
Honsiger, et al., 1991	sandstone, 2"x2"x4" (5x5x10cm)	various	partial immersion of 5x5cm side in 0.5cm product	1 month	(not noted)	
Lukaszewicz, 1994	tuff, 1.6"x0.4"x3.1" (4x1x8cm)	Wacker OH	partial immersion: placed 1x8cm side in 1cm of product for 24 hours	6 weeks at 75% RH, then longer at 40-50% RH	full penetration	
Wheeler, et al., 1992	marble, 0.25" (6.3mm) diameter x 2.4" (60mm) long cylinders	various	partial immersion: half length immersed for 45 minutes	2 weeks	full penetration	
Applonia, 1995	various types, 2"x2"x0.8" (5cmx5cmx2cm)	various	capillary absorption on multi-layer bed of filter paper soaked in product, 6-24 hours	30 days	not reported	tested amount of product absorbed 1 month and 1 year after application was complete; showed that temperature and RH affect amount of product absorbed and polymerization rate of silicon-based products
De Witte, et al., 1985	Belgium limestone, 2"x2"x4" (5cm x 5cmx10cm)	various available in Belgium	capillary absorption from foam soaked in product	wrapped all surfaces in foil (except that in contact with foam), cure 2 weeks	poor penetration of epoxy, 1.5-2.4" (40-60mm) with organosilicium compounds	used microdrop absorption test to measure impregnation depth
Lukaszewicz, et al., 1995	Gotland sandstone	Wacker OH	capillary suction	suggests covering surface with foil; time not noted; indicated temperature of 50-68°F (10-20°C) and RH above 40% is best	(not noted)	treated samples darkened; reports penetration of 4-8" (10-20 cm) is possible
Weber, 1987	conglomerate stone, building façade	Wacker OH	saturating spray	3 weeks (assumed)	40-65 mm	consumption was 1.2 l/m²
Wihr, 1976	sandstone sculpture	Wacker OH	continuous spray in polyethylene sheeting for several hours	not reported	5cm per hour	is a patented method
Tabasso, et al., 1994	tuff, 10.6" (27 cm) cubes	various (including OH)	needle infusion for 4-7 days (see notes)	21 days	not recorded, but based on compressive strength	specimen wrapped in gauze-wool layer, place on supports in tank, needles placed in gauze allowed to drip slowly
Selwitz, 1992	Weathered sandstone, building facade	Epoxy resin in various solvents	pocket method	not reported	9-10 cm	pockets were difficult to prepare, leaked and left marks on stone
Caselli, et al., 1995	Australian sandstone, various sizes	various (including OH)	5 flood applications with squeeze bottle (see notes)	flush surface with white spirits, cure 2 months	1.2"-1.6" (30-40mm) with OH	flood applications 5-10 minutes apart; 20-30 cycles of this with 40 min between cycles; used microdrop absorption test to determine penetration

Table 6.9

Determining Depth of Penetration of Consolidant: Literature Review

Depth of penetration depends on the nature of the substrate (namely, permeability), the composition and viscosity of the consolidant, and the way in which the consolidant is applied to the substrate. In a laboratory setting, chemical or visual/mechanical means can be used to determine depth of penetration on an exposed cross-section taken from the specimen. In the field, non-destructive tests (usually ultrasonic pulse velocity measurements) can be used in lieu of taking core samples from the building façade.

Chemical and visual/mechanical means of determined penetration depth include the following:

- observing grain structure and presence of consolidant with a microscope or SEM
- placing the cross-section in an iodine rich environment to stain the specimen
- adding dyes to the consolidant at the time of application to trace its penetration
- heating the specimen to char organic resins
- use of spectroscopy to find functional groups or elements associated only with the consolidant
- measuring contact angle of water droplets placed on the cross-section
- measuring the absorption time of a water droplet on the cross-section.

Microscopy and spectroscopy were ruled out as means of determining penetration depth because the structures of the OH and concrete are thought to be too similar. The iodine crystal method was ruled out because it has been shown by others to be inconclusive with ethyl silicate consolidants. Dye methods can also be inconclusive since low-viscosity dyes can be carried farther into the substrate than the consolidant.

Measuring contact angle or absorption time of a water droplet on the treated surface is based on the fact that many consolidants are somewhat hydrophobic. On a hydrophobic surface, the contact angle and absorption time of a water drop will be greater than that on an untreated surface. Although OH is described as a non-hydrophobic product, some researchers have found that these methods are appropriate for this consolidant. In fact, Weber and Zinsmeister state, "Within the first 14 days after application, hydrolysis of tetraethyl silicates is not yet complete; a small quantity of residual organic groups is responsible for a moderate water-repellency effect, which disappears in the following weeks."[20]

Because equipment to properly measure contact angle was not available for this study, RILEM's "Water Drop Absorption Test" (also called the "microdrop absorption test" because the water droplets used are quite small) was used to determine the penetration dept of OH into the concrete specimen. This method relies on the hydrophobicity of OH during polymerization. The amount of water and the rate of absorption presumably directly depend on the amount of product in the substrate.[21] Thus, microdrop absorption data provide a way to assess the distribution of the consolidant in the substrate.

---

[20] *Conservation of Natural Stone: Guidelines to Consolidation, Restoration and Preservation*, 78.
[21] M. Laurenzi Tabasso, A.M. Mecchi, and U. Santamaria, "Interaction Between Volcanic Tuff and Products Used for Consolidation and Waterproofing Treatment," in *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990* (Rome: ICCROM, 1994), 183-184.

113
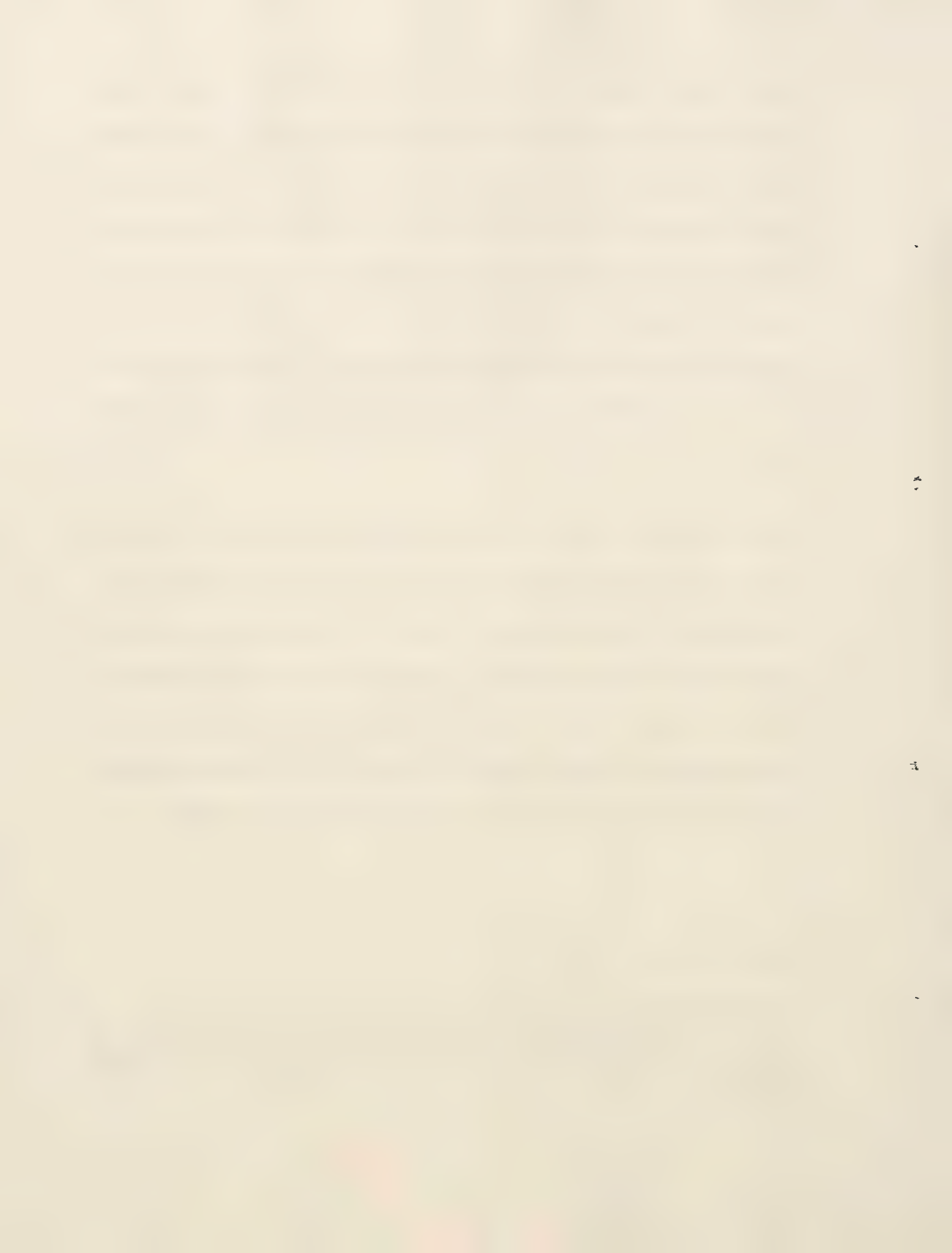
To reveal cross-sections for the microdrop test, untreated and treated cubes were cut in half with a masonry saw lubricated with water. A hand-held drill with a masonry coring bit was used to take cores from the cylinders. Tap water was used to lubricate the bit during coring. The bit yielded cores approximately 0.875" (2.2 cm) in diameter and approximately 1.875" (4.8 cm) long. The cores were then cut longitudinally with a masonry saw lubricated with water to reveal a flat cross-section for the microdrop absorption test. On the cube and core cross-sections, microdrops were placed in a line perpendicular to the exterior treated surface, starting from the treated surface and moving inward. Figure 6.6 illustrates the drop placement on the cubes. Drops were placed on the cylinder cores in a one straight line every 1/4" (6.35 mm) from the treated surface inward. Appendix C describes the complete procedure for the microdrop absorption test.

Placement of Microdrops on Cross-Section of Cubic Specimen

Figure 6.6

114
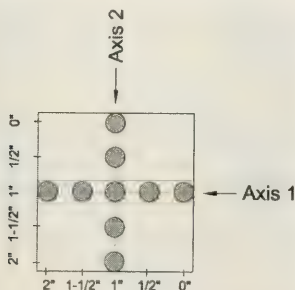
Application of Conservare OH to Concrete Specimen

Cubes: Treatment for Preliminary Testing

Preliminary testing was completed to determine if the Conservare OH could fully penetrate the 2" concrete cubes via total immersion. The cubes from Batch A were used for preliminary testing. 0.25" (6 mm) glass beads were placed in the bottom of a plastic container. The cubes were placed on top of the beads, and the Conservare OH was poured over the cubes.[22] Approximately 0.4" (1 cm) of liquid was maintained over the top surface of the cubes during the treatment duration. In the preliminary tests, the immersion containers were not covered.

In order to determine suitable immersion duration, one of the cubes was immersed for three days, one was immersed for four days, and three were immersed for five days. After twenty-three days of curing, the microdrop absorption test was done to determine penetration depth of the OH. Appendix C includes the raw data collected during testing. The results are discussed in detail in Appendix C. The preliminary test results are summarized in Table 6.10 and are presented graphically in Figure 6.7. During the test, it was often observed that the microdrops did in fact "bead" on the treated surfaces, and that they did not bead or beaded to a lesser extent on the untreated specimen.

---

[22] The OH was used as-is; that is, it was not diluted. A 5-gallon drum of the product was purchased. The lot number was 2753-0799.

Preliminary Microdrop Absorption Test:
2" Concrete Cubes

Average Microdrop Absorption Time (minutes)						
Treatment	0"	1/2"	Center	1-1/2"	2"	Average
Untreated	1.03	0.95	2.04	1.22	0.84	1.21
Immersed 3 days	1.72	2.03	2.07	1.76	2.04	1.92
Immersed 4 days	2.19	1.74	2.10	1.82	2.05	1.98
Immersed 5 days	2.76	2.27	2.44	2.45	2.69	2.52

Table 6.10

Preliminary Microdrop Absorption Test:
2" Concrete Cubes
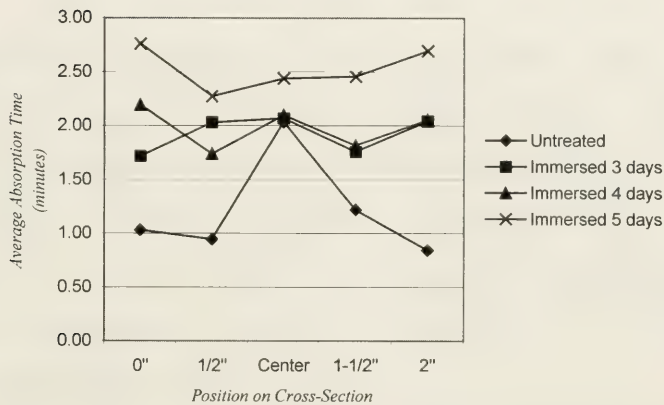
Figure 6.7

Analysis of variance between the untreated specimen and the pooled treated specimen proved that there is a statistically significant difference in microdrop absorption times of the untreated specimen and the treated specimen. This proves that the treated specimen are in fact *treated*. The F-tests shown in Appendix C indicate that there is no statistically

116

significant difference in absorption time from one 1/2" position to the others on one specimen; that is, on an individual specimen, the 0" position time does not differ from the 1/2", does not differ from the center, etc. This is true for the specimen immersed for three days, for the one immersed four days, and for the one immersed for five days. This indicates that the treated specimen are statistically *fully impregnated*. Analysis of variance between the 3-day, 4-day and 5-day treated specimen showed that there is no statistically significant difference between them. This leads one to believe that immersing the specimen for three days is as effective as immersing them for four or five days. The average absorption times reported in Table 6.10 show an increase in absorption time from the untreated specimen to the 5-day treated specimen, with an approximate 0.70 minute increase from the untreated specimen to the 3-day treated specimen. The average absorption time also increases from 3-day to 4-day to 5-day treatment. This could indicate that although the 3-day treated specimen are fully impregnated, the 4-day and 5-day treated specimen are fully impregnated and contain more consolidant (as expected).

Cubes: Treatment for Final Testing

The results of the preliminary microdrop absorption test indicated that the full immersion technique is effective in treating and fully impregnating the cubic concrete specimen. As was the objective of immersing the specimen, pure could be produced with this treatment technique. Thus, treatment of cubic specimen from Batches B, D and F was done for final testing. Six cubes from Batch B, seven cubes from Batch D, and five cubes from Batch F were immersed in Conservare OH for five days. Two cubes from Batch F were

117

immersed for one hour for comparison. The specimen were elevated from the bottom of the plastic container by glass beads and 0.4" (1 cm) of liquid remained over the top of the cubes for the duration of immersion; for this treatment, the plastic containers were tightly covered to deter solvent evaporation and prolong gelation time. After treatment, the cubes were brushed with methyl ethyl ketone (MEK) to eliminate surface crust formation, as recommended by ProSoCo.

Two of the untreated specimen were taken for baseline absorption time, and six of treated specimen was taken to determine depth of penetration of the treatment (ten days after treatment). Appendix C provides the raw data collected during this final testing. The results are discussed in detail in Appendix C. The final test results are summarized in Table 6.11 and are presented graphically in Figure 6.8.

Final Microdrop Absorption Test:
2" Concrete Cubes

Average Microdrop Absorption Time (minutes)					
Treatment	0"	1/2"	Center	1-1/2"	2"
Untreated	1.55	1.54	1.44	1.61	1.42
Immersed 1 Hour	1.97	1.24	1.35	1.21	1.77
Immersed 5 Days	2.88	2.24	1.48	1.76	2.43

Table 6.11

Final Microdrop Absorption Test:
2" Concrete Cubes
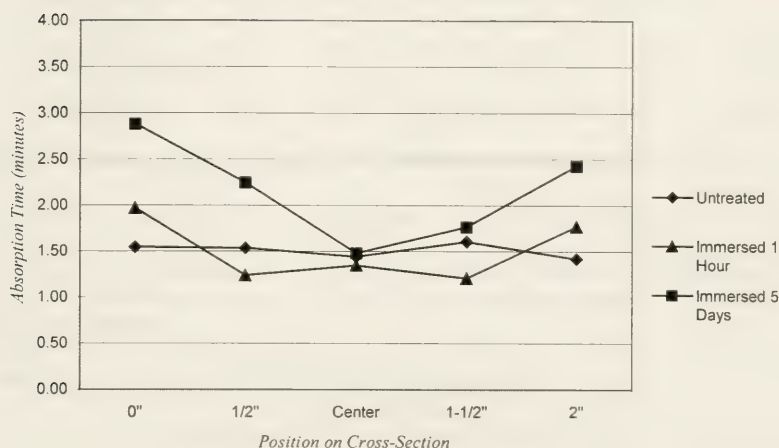
Figure 6.8

Prior to statistical analysis, the average microdrop absorption times seem to indicate that there very little difference between the untreated and 1-hour treated specimen. Furthermore, the average absorption times of the preliminary test and the final test are similar (compare Tables 6.10 and 6.11). Figure 6.8 shows that some consolidant penetrated the very edges of the 1-hour treated specimen, but that consolidant was unable to deeply penetrate the specimen in such a short immersion time. The graph indicates that deeper penetration was achieved in the specimen treated for five days. Analysis of variance between the untreated and 5-day treated specimen indicates that there is a statistically significant difference in absorption times between the treated and untreated specimen. This is indicates that the treated specimen are in fact *treated*.

119

The F-test, however, indicates that there is a statistically significant difference in absorption time between the center and edge positions on the 5-day treated specimen. Figure 6.8 shows the absorption times of the center positions of the untreated and 5-day treated specimen are almost the same. This indicates that the 5-day treated specimen may not be fully impregnated. To assess the depth of penetration more closely, absorption times were recorded for the 3/4" and 1-1/4" positions on both axes of the 5-day treated cross-sections (but not recorded on the untreated or 1-hour treated specimen, nor retimed at the original 1/2" positions of the 5-day specimen). Table 6.12 and Figure 6.9 summarize the results.

Final Microdrop Absorption Test:
2" Concrete Cubes
Including the 3/4" and 1-1/4" Positions

Average Microdrop Absorption Time (minutes)							
Specimen	0"	1/2"	3/4"	Center	1-1/4"	1-1/2"	2"
Untreated	1.55	1.54		1.44		1.61	1.42
Immersed 1 Hour	1.97	1.24		1.35		1.21	1.77
Immersed 5 Days	2.88	2.24	2.72	1.48	2.54	1.76	2.43

Table 6.12

The absorption times at the 3/4" and 1-1/4" positions were higher than those at the 1/2" and 1-1/2" positions. This was unexpected; it was expected that the resulting absorption time curve would decrease more gradually toward the center position, giving a flatter absorption time curve. Measuring the absorption time at the 3/4" and 1-1/4" positions occurred approximately one week after the initial 1/2" position readings; this delay may have skewed the results somewhat, as the OH could have polymerized further in all positions during the week between tests.

120

Final Microdrop Absorption Test:
2" Concrete Cubes
Including the 3/4" and 1-1/4" Positions
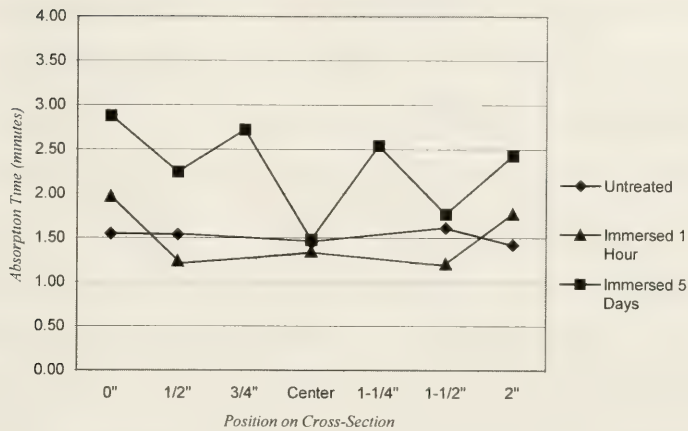
Figure 6.9

It is apparent that the OH *did not fully impregnate* the cubes from Batches B, D, and F. The reason for the discrepancy in penetration of Batch A and the later batches is not known; in fact, because the water/cement ratio of Batches B-G was higher than that of Batch A, it was expected that the later batches would be more porous and permeable. If consolidant only penetrated 3/4" (1.9 cm) in from all outside surfaces of the cubes (leaving a circle of 1/2" [1.27 cm] diameter in the center of the cube), then 3.8 in^2 (24.5 cm^2) of the cross-section is treated. This is actually at least 95% of the cross-section. For the purposes of this study, it will be assumed that the treated 2" cubes were fully impregnated.

<u>Cylinders: Preliminary Application Trials</u>

The objective of treating the cylinders was to obtain a composite material, that is, one with a treated outer shell and an untreated inner core. Two of the cylinders from Batch A were chosen for brush application tests. ProSoCo recommends that brush application be done in three cycles: each cycle consists of three saturating applications spaced five to fifteen minutes apart, with twenty to sixty minutes between each cycle. Although full impregnation of the cylinder's cross-section was not desired, at least 1" (2.54 cm) penetration was desired. Preliminary treatment trials were done to test the application procedure described by the manufacturer. The consolidant readily soaked into the concrete during the first application of the first cycle. The same was observed for the second and third applications of the first cycle. During the five minutes between applications, the consolidant completely soaked into the concrete. Twenty minutes was allowed between cycles. During the second and third cycles, the surface became glossier, and it took the consolidant longer to soak in between applications. Between all cycles, however, the consolidant was completely absorbed by the concrete. A fourth cycle was then applied to the surface. Twenty minutes after the cycle was complete, the surface felt "greasy" as if not all the consolidant had soaked in. A fifth cycle was applied with similar results. Thus, it was decided to use the manufacturer's recommended three-cycle application technique. This is a valuable laboratory approach, since this technique is probably specified and used in the field. No cores were taken from the Batch A cylinders to determine penetration depth of the preliminary treatment.

<u>Cylinders: Treatment for Final Testing</u>

The three-cycle brush application technique was used to treat some of the cylinders from Batches B-G. Only the 12" height of the cylinder was treated, as the 6" circular top and bottom were capped. Five minutes was allowed between each OH application, and twenty minutes was allowed between cycles. Twenty minutes after the last application of the third cycle, methyl ethyl ketone was brush-applied to the treated surfaces. Both the untreated and treated cylinders cured in ambient lab conditions after treatment.

Appendix C provides the raw data collected from the microdrop absorption test on the cylinder cores. The results are discussed in detail in Appendix C and are summarized in Table 6.13 and Figure 6.10.

Microdrop Absorption Test:
Concrete Cylinder Cores

Average Microdrop Absorption Time (minutes)							
Treatment	0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
Untreated	1.58	2.61	3.53	2.76	3.91	2.86	3.26
Brush application	6.22	4.92	5.36	3.85	4.37	3.00	2.80

Table 6.13

The absorption time curve in Figure 6.10 indicates at least 3/4" and possibly 1" penetration of the OH treatment. An F-test was used to compare the average absorption times at positions along the untreated cores and those along the treated cores. Although no statistically significant difference between the edge, 1/4", and 1/2" positions of the untreated and treated cores was seen, there was a statistically significant difference between the 3/4" and 1" positions of the untreated and treated specimen. When

123

comparing one treated position to the other treated positions, there was no statistically significant difference observed. Taking into account both of these F-tests and the graphic illustration in Figure 6.10, it was determined that the OH penetrated 1" into the concrete cylinders.

Microdrop Absorption Test:
Concrete Cylinder Cores
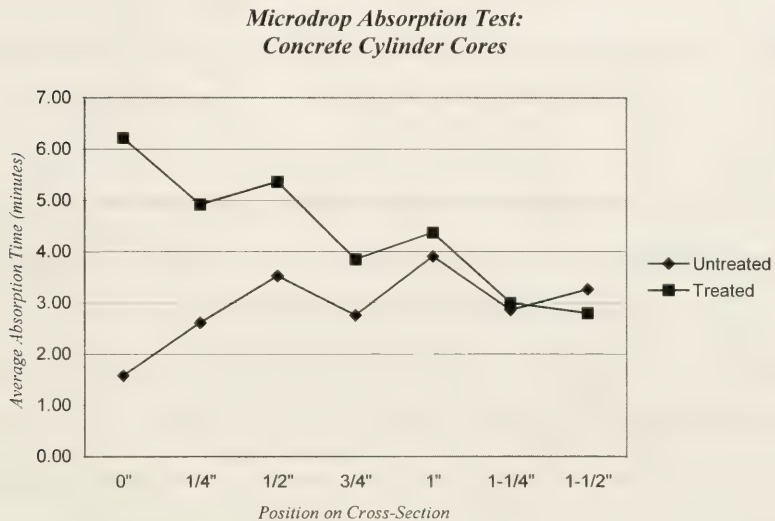
Figure 6.10

Curing the Untreated and Treated Specimen

Curing Conditions and Observations of the Cubes

The cubes were removed from their molds twenty-four hours after forming and were then placed into tightly sealed plastic containers for five days. After four days in the plastic containers (five days after batching), the surfaces were rough-polished in preparation for eventual compression testing. The cubes were then replaced into the sealed plastic

124

containers. Two days later, those cubes chosen for treatment were immersed in the OH. While these cubes were immersed, the untreated cubes were left in the sealed containers. When treatment was complete, both the treated and untreated cubes cured in open air in ambient lab conditions of 63±5°F (17.2°C) and 35±5% RH. The treated cubes were placed on a baker's cooling rack so that excess OH and MEK could freely drip away from the surface. After treatment, the cubes cured for approximately twenty-eight days before compression testing.

All of the cubes were weighed prior to treating the selected cubes (see raw data in Appendix D). The average density of the cubes prior to treatment was 153.4 lb/ft^3 (2.44 g/cm^3). The treated cubes absorbed an average of 0.003 lb (1.38 g) of consolidant such that they experienced an average 0.43% weight increase after treatment. Although it seems that this is a small amount of consolidant to be absorbed in treatment, the concrete weighs a lot itself and is quite dense. An F-test indicated that there was no statistically significant difference between Batches B, D, and F.

It was desired to know if the untreated cubes cured differently than the treated cubes. All of the cubes were weighed periodically during the curing period (see raw data in Appendix D). From these measurements, weight change from seven days after batching (when some of the specimen were treated) until forty days after batching (the day of compression testing) was examined. The weight change of the untreated cubes is graphed in Figure 6.11, and that of the treated cubes is graphed in Figure 6.12. Weight gain (positive weight change) shown in Figure 6.12 indicates the average amount of

Weight Change after Batching:
Average of Each Batch of Untreated Cubes
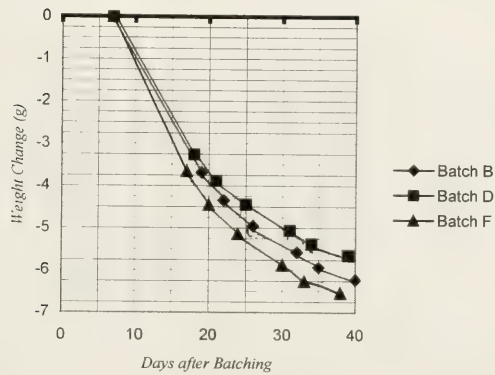
Figure 6.11

Weight Change after Batching:
Average of Each Batch of Treated Cubes
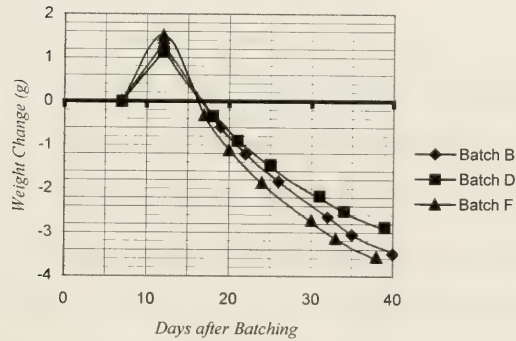
Figure 6.12

consolidant absorbed by the specimen. Weight loss (negative weight change) is attributed to water loss during hydration of the concrete in both untreated and treated specimen.

In both the untreated and treated cubes, Batch B lost less weight than did Batch D, and Batch D lost less weight than did Batch F. This is attributed to minor differences in the preparation of the batches. The slopes of the weight change curves for the untreated and treated cubes are very nearly the same. The untreated specimen lost an average of 6.2 g. The treated specimen absorbed an average of 1.38 g of consolidant, then lost an average of 3.3 g. The average gross change in weight of the treated cubes was thus 4.68 g, which is 1.5 g less than the gross change of the untreated cubes. This could indicate the amount of water used in the hydrolysis of the ethyl silicate. Although the cubes did not cure to constant weight, time constraints mandated that the specimen undergo compression testing before constant weight was achieved.

When the cubes were taken out the immersion bath, they appeared much darker (browner) than the untreated cubes. Although this darkening lesssened during curing, after twenty-eight days, the treated cubes were still darker than the untreated cubes (see Figure 6.13). In addition, the darkening was not even over the surface such that the surface appeared mottled. Other researchers have shown little to no color change in stone treated with ethyl silicates.
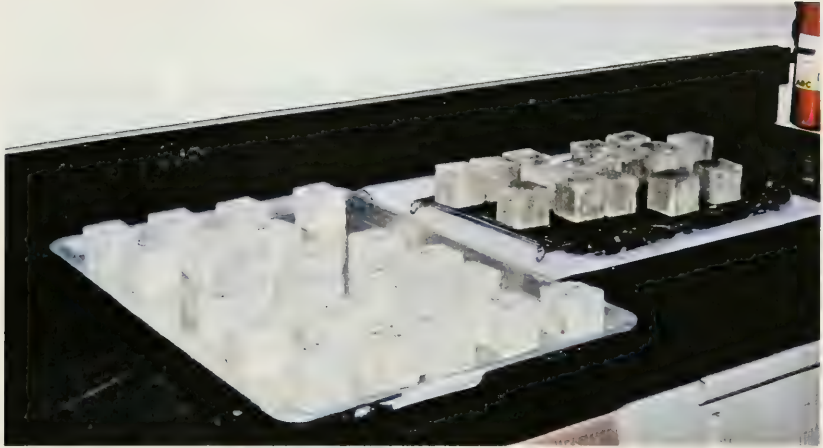
Figure 6.13

Note darkened and mottled appearance of the treated cubes on the right.

Curing Conditions and Observations of the Cylinders

The cylinders were removed from their molds twenty-four hours after forming and were placed into polyethylene trash bags. After four days in the bags (five days after batching), all of the cylinders were removed from the bags and some of the cylinders were capped. Due to lab restrictions, the rest of the cylinders had to be capped two days later. After treating some of the cylinders, the untreated and treated cylinders were left to cure in open air in ambient lab conditions of 63±5°F (17.2°C) and 35±5% RH. The treated cylinders were placed on baker's cooling racks to allow excess OH and MEK freely drip away from the surface. After treatment, the cylinders cured approximately

twenty-eight days before compression testing. The cylinders were not weighed to determine amount of consolidant absorbed or to monitor curing differences.

Like the cubes, the treated surfaces of the cylinders also darkened after treatment. After the curing period, however, the color difference between untreated and treated cylinders was less pronounced than that seen with the cubes. One explanation for this observation could be the fact that the cylinders were in contact with the treatment for a shorter duration.

CHAPTER SEVEN: TESTING RESULTS AND ANALYSIS

Compression Testing: Introduction

In ASTM's "Guidelines for the Selection and Use of Stone Consolidants" it is stated, "Compressive strength measurements are important if the consolidated stone is required to perform a load-bearing function."[1] As shown in Chapter Five, increased strength allows additional load to be placed on structural materials. The untreated and treated cubes and the composite cylinders prepared for this thesis were subjected to compression loading to examine any differences in the strength of the different specimen types. Stress-strain data collected during compression testing was interpreted to determine the modulus of elasticity of the specimen. Compression testing results of concrete specimen are only comparative within the specimen and indicate the quality and strength of the concrete mix used. No exact relationship exists between compressive strength and modulus of elasticity.[2]

ASTM C39 "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimen," ASTM C109 "Compressive Strength of Hydraulic Mortars (Using 2-in. or 50mm Cube Specimens)", and RILEM's "Ultimate Compressive Strength, Test No. III.5" were used as standard methods for measuring compressive strength. ASTM recommends keeping concrete specimen wet (in moist room or under water) until the time of

---

[1] Draft No. 11, September 1998.
[2] Derucher, et al., *Materials for Highway and Civil Engineers,* 3rd ed., 107.
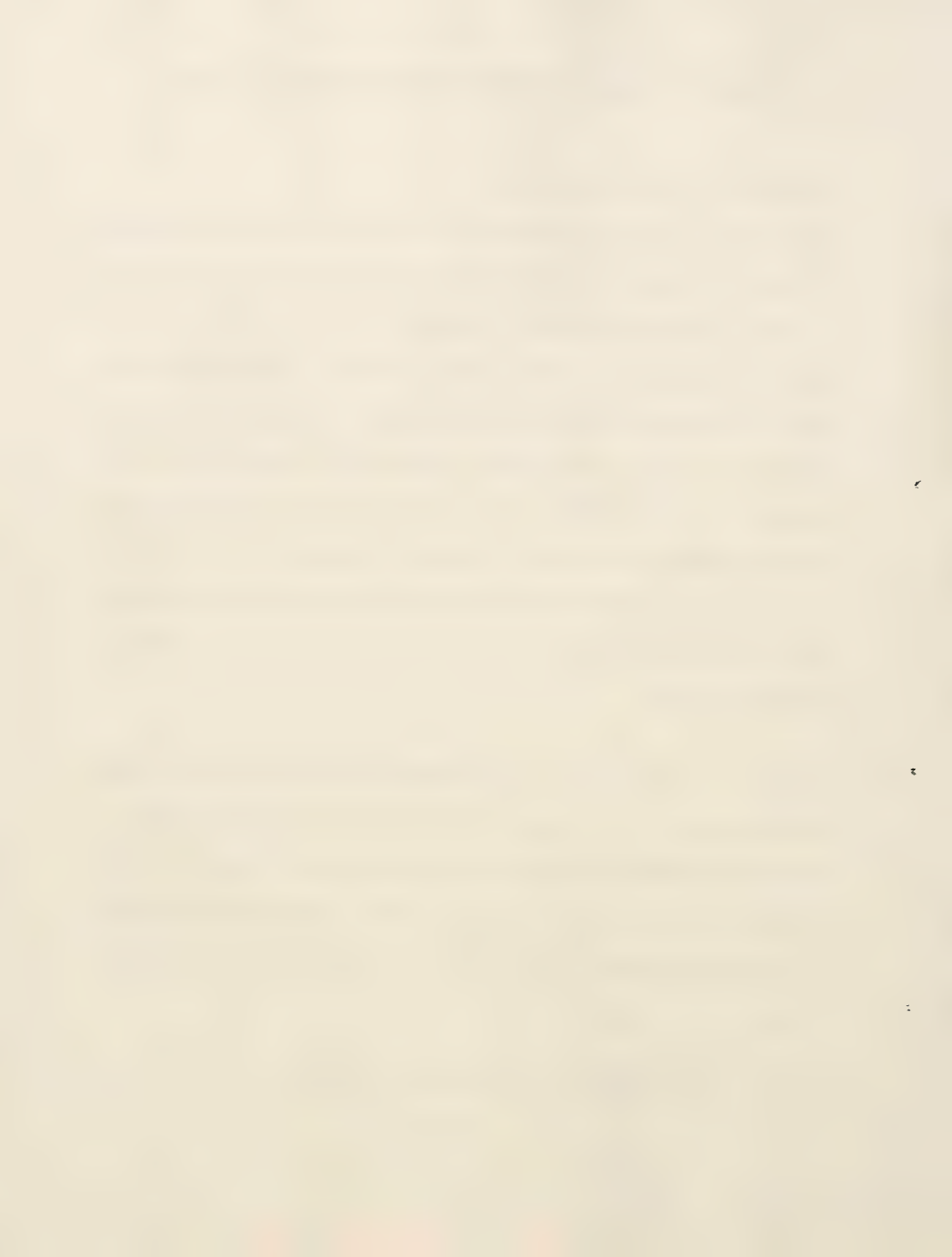
compression testing. RILEM specifies use of no fewer than three test specimen, which should be dry before testing. Testing wet material usually results in lower compressive strength than when testing dry material. RILEM's standard is written primarily for stone. The concrete specimen used in the testing for this thesis were not wet-cured, nor were they dried before testing. Because the curing and testing environments of the cubic and cylindrical specimen were the same, however, comparisons between their test results can be made. Comparing the results from this thesis to others' work (with different curing and testing conditions) should not be directly made.

ASTM C469 provides a standard for determining the static modulus of elasticity of concrete in compression. This method specifies use of a compressometer, which is a sensing device attached to the specimen during loading. A compressometer was not available for use during testing for this thesis. Instead, a dial gage was used to record relative displacement of the bearing plates (and thus the ends of the specimen) during compression.

Derucher, et al., show that concrete exhibits linear-elastic behavior up to about 45% of its maximum compressive strength.[3] Walsh notes that as microcracks close with increasing uniaxial compression, the specimen becomes stiffer to linearity.[4] Nonlinear behavior after this stress level causes propagation and sliding of the microcracks, especially at

---

[3] *Materials for Highway and Civil Engineers,* 3rd ed., 101+.
[4] "Deformation and Fracture of Rock," 94.

interfaces between the aggregates and cement paste. The stress-strain curve shows a gradual increase in curvature up to about 95% of the maximum compressive strength.

A line joining the origin of the stress-strain curve (load = $\sigma = 0$, and displacement = $\varepsilon = 0$) to any point on the curve gives the "secant modulus of elasticity." The value of the secant modulus of elasticity at $0.45\sigma_{c.max}$ is generally used as the modulus of elasticity of the concrete specimen. ACI offers the following equation to predict E:

$E = 33w_c^{1.5}\sqrt{\sigma_c}$, where w_c is the density of the concrete in pcf and E is reported in psi.[5]

Compression Testing Results

Cubes from Batch A were used in preliminary tests to obtain a rough idea of the compressive strength and modulus of elasticity of the specimen. The preliminary tests were done at the Laboratory for Research on the Structure of Matter (LRSM) at the University of Pennsylvania. LRSM was conveniently located near the treatment lab, and had equipment suited to the 2" specimen size. The final testing of cubes and cylinders from Batches B-G was done at Pennoni Associates Inc., in Bridgeport, Pennsylvania. This lab had larger equipment for testing the cylinders. Testing equipment at both labs was set up for U.S. units. The results were then converted to metric units.

---

[5] *Building Code Requirements for Structural Concrete and Commentary (ACI 318-95)* (Farmington Hills, MI: American Concrete Institute, 1995).

Preliminary Results

Table 7.1 records the preliminary testing conditions.

Preliminary Compression Testing Conditions

Preliminary Testing Location	LRSM, University of Pennsylvania
Loading Equipment	Instron Testing Machine 4206 (capacity 30,000 lb)
Load/Displacement Rate	0.01 inch/minute
Load Recordation Device	digital display with pen plotter
Displacement Recordation Device	digital display with pen plotter
Ambient Lab Conditions	68±5°F (20 °C)
Testing Date	17 January 2000
Time elapsed from batching to testing	Batch A: 55 days

Table 7.1

Appendix E includes the results of the testing and the load-displacement graph obtained during preliminary compression loading of some of the Batch A specimen.   Tables 7.2a (U.S. units) and 7.2b (metric units) summarize the results.

Preliminary Compression Test: 2" Cubes (U.S. Units)

Specimen	Avg. Maximum Load (lb)	Avg. Maximum Compressive Stress (psi)	E (ksi)
Untreated 2" Cubes	22,610	5652.5	210.3
Treated 2" Cubes	22,590	5647.5	217.3

Table 7.2a

Preliminary Compression Test: 5 cm Cubes (Metric Units)

Specimen	Avg. Maximum Load (N)	Avg. Maximum Compressive Stress (MPa)	E (GPa)
Untreated 2" Cubes	101,745	39.0	1.55
Treated 2" Cubes	101,655	38.9	1.50

Table 7.2b

133

The average compressive strength of the untreated cubes from Batch A was 5652.5 psi (39.0 MPa). The average compressive strength of the treated cubes from Batch A was 5647.5 psi (38.9 MPa). The results of the preliminary test indicate that the untreated and treated cubes did not have a significantly different compressive strength. The compressive strength of the concrete is higher than most stones (see Tables 5.1a and 5.1b).

The average modulus of elasticity of the untreated cubes from Batch A was 210.3 ksi (1.55 GPa). The average modulus of elasticity of the treated cubes from Batch A was 217.3 ksi (1.50 GPa). The modulus of elasticity was not significantly different between the treated and untreated specimen. The modulus of elasticity of the concrete was much lower than expected. The average density of the 2" concrete samples before treatment was 153 pcf (2451 kg/m^3). Using the ACI equation for approximating E (w_c = 153 pcf, σ_c = 5650 psi), the expected E for the specimen was 4690 ksi. This indicates E for the specimen deviated from the expected value by one order of magnitude. A quick retest of a specimen proved that the modulus was computed correctly and that the testing equipment constants were correctly recorded. The reason for the lower-than-expected modulus of elasticity found in the preliminary testing is unknown.

The sample size for the preliminary tests was quite small: only three untreated cubes and three treated cubes were tested. Because the sample size was so small, no statistical analysis was performed for the preliminary tests. The preliminary tests were not meant to be conclusive, only to direct final testing of a larger sample group.

134

Final Results

Table 7.3 records the final testing conditions. Appendix E includes the data obtained during final compression loading of the cubes and cylinders and the results of the testing. The maximum load and relative displacement (compression) of the specimen were recorded. The data was then graphed by the author to provide information on the load-deformation behavior of the specimen during testing. One of the twelve untreated cubes, one of the thirteen untreated cylinders, and one of the thirteen treated cylinders did not show linear behavior during compression. The results from those specimen not showing linear behavior at some point in the loading were not included in the statistical analysis of the results. Non-linear behavior could have been due to non-uniform loading rate or to a material flaw within the specimen.

Final Compression Testing Conditions

Final Testing Location	Pennoni Associates, Inc., Bridgeport, PA
Loading Equipment	Cubes: Tatnall Measuring Systems Compression Machine Cylinders: Baldwin Compression Machine
Load/Displacement Rate	Cubes: approx 350 lb/sec (90 psi/sec) Cylinders: approx 2000 lb/sec (88 psi/sec)
Load Recordation Device	Analog dial gage (increments of 250 lb)
Displacement Recordation Device	Cubes: Starrett Analog Dial Gage (to 0.001") Cylinders: CDI Analog Dial Gage (to 0.001")
Ambient Lab Conditions	63±5°F (17 °C)
Testing Date	22 February 2000
Time elapsed from batching to testing	Batches B & C: 40 days Batches D & E: 39 days Batches F & G: 38 days

Table 7.3

Cubes

The data and analysis from the compression testing of the cubes is included in Appendix E. The larger sample size of the final testing group helped to reduce standard deviation and confidence interval, but scatter is still evident in the results. F-tests proved that there was no statistically significant difference between the results of the cubes from Batches B, D, and F for either load/stress or modulus of elasticity. Thus, the results from the three batches were pooled to analyze the differences between the untreated and treated pools. Tables 7.4a (U.S. units) and 7.5b (metric units) summarize the results with 90% confidence intervals. Figure 7.1 is a sample load-displacement curve constructed from the data obtained during compression of an untreated cube. Figure 7.2 is the curve of a treated cube.

Final Compression Test: 2" Cubes
(U.S. Units)

Specimen	Avg. Maximum Load (lb)	Avg. Maximum Compressive Stress (psi)	E (ksi)
Untreated 2" Cubes	22,000 ± 1208	5500 ± 302	734 ± 116
Treated 2" Cubes	25,250 ± 1677	6313 ± 401	1095 ± 212

Table 7.4a

Final Compression Test: 5 cm Cubes
(Metric Units)

Specimen	Avg. Maximum Load (N)	Avg. Maximum Compressive Stress (MPa)	E (GPa)
Untreated 5 cm Cubes	99,000 ± 5436	37.9 ± 2.1	5.1 ± 0.8
Treated 5 cm Cubes	113,625 ± 7547	43.5 ± 2.8	7.6 ± 1.5

Table 7.4b

136

Sample Load-Displacement Curve:
Untreated Cube (Fb6)
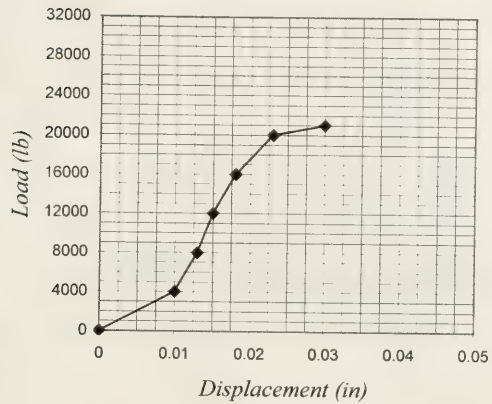
Figure 7.1

Sample Load-Displacement Curve:
Treated Cube (Fb3)
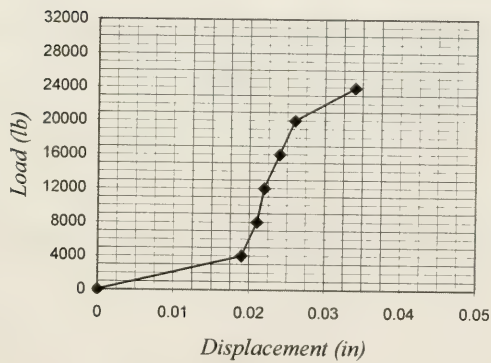
Figure 7.2

The difference between the average value of compressive load of the untreated and treated cubes is 3250 lb (14,625 N). Analysis of variance (included in Appendix E) showed that there is a statistically significant difference between the two treatments' maximum compressive load. Treatment increased the maximum load sustained by the concrete cubes by 12.9%.

The difference between the average maximum compressive stress of the untreated and treated cubes is 813 psi (5.6 MPa). Analysis of variance showed that there is a statistically significant difference between the two treatments' maximum compressive stress. Treatment increased the maximum stress sustained by the concrete cubes by 12.9%.

The difference between the average modulii of elasticity for the untreated and treated specimen is 361 ksi (2.5 GPa). Analysis of variance showed that there is a statistically significant difference between the treatments' modulii of elasticity. Treatment increased the maximum load sustained by the concrete cubes by 33%. As was the case in the preliminary testing, the modulii of elasticity for the untreated and treated cubes were lower than is expected for concrete.

The results will be discussed and analyzed in more depth later in this chapter.

Cylinders

The data and analysis from the compression testing of the cylinders is included in Appendix E. F-tests proved that there was no statistically significant difference between the results of the cylinders from Batches B-G for either load/stress or modulus of elasticity. The results from the five batches were pooled to analyze the differences between the untreated and treated pools. Tables 7.5a (U.S. units) and 7.5b (metric units) summarize the results with 90% confidence intervals. Figure 7.3 is a sample load-displacement curve constructed from data obtained during compression of an untreated cylinder, and Figure 7.4 is the curve of a treated cylinder.

Final Compression Test: 6"-diameter Cylinders
(U.S. Units)

Specimen	Avg. Maximum Load (lb)	Avg. Maximum Compressive Stress (psi)	E (ksi)
Untreated 6" Cylinders	142,583 ± 4898	5044 ± 173	2210 ± 151
Treated 6" Cylinders	151,295 ± 6340	5352 ± 224	2352 ± 276

Table 7.5a

Final Compression Test: 15.25 cm-diameter Cylinders
(Metric Units)

Specimen	Avg. Maximum Load (N)	Avg. Maximum Compressive Stress (MPa)	E (GPa)
Untreated 15.2 cm Cylinders	641,619 ± 22,041	34.8 ± 1.2	15.2 ± 1.0
Treated 15.2 cm Cubes	680,828 ± 28,530	36.9 ± 1.5	16.2 ± 1.9

Table 7.5b

Sample Load-Displacement Curve:
Untreated Cylinder (Ec1)
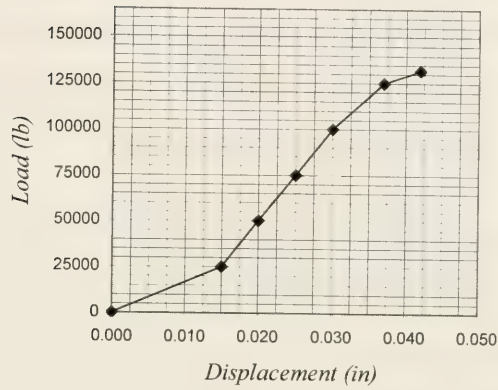
Figure 7.3

Sample Load-Displacement Curve:
Treated Cylinder (Cc5)
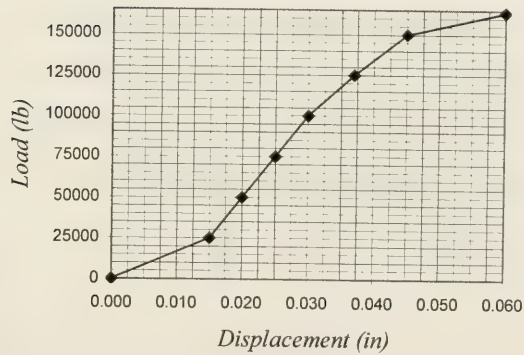
Figure 7.4

140

The difference between the average value of compressive load of the untreated and treated cylinders is 8712 lb (39,204 N). Analysis of variance showed that there is a statistically significant difference between the two treatments' maximum compressive load. Treatment increased the maximum load sustained by the concrete cylinders by 5.8%. Because they are larger, the cylinders withstand a higher maximum compressive load than the cubes.

The difference between the average maximum compressive stress of the untreated and treated cubes is 308 psi (2.1 MPa). Analysis of variance showed that there is a statistically significant difference between the two treatments' maximum compressive stress. Treatment increased the maximum stress sustained by the concrete cylinders by 5.8%. Comparing the untreated cubes with the untreated cylinders shows that the average maximum stress in the cubes (5500±302 psi) is slightly higher than that of the cylinders (5044±173 psi). Several theories could explain this phenomenon, including that which takes into account the size of the specimen: larger specimen have larger "flaws" resulting in impeded performance. It is interesting that most researchers often use small (2" or 5 cm) specimen. The fact somewhat higher (8% in this case) maximum stress can be observed with smaller specimen should be always discussed in research studies.

The difference between the average modulii of elasticity for the untreated and treated specimen is 142 ksi (1.0 GPa). Analysis of variance showed that there is *not* a statistically significant difference between the treatments' modulii of elasticity. The

141

modulii of elasticity of the final specimen were closer to the value expected by the theoretical value calculated from the ACI equation. Comparing the average modulus of elasticity of the untreated cubes and the untreated cylinders shows that E of the cylinders is much higher (67%) than that of the cubes. The 12" (30.5 cm) cylindrical specimen are taller and can sustain higher loads than the 2" (5 cm) cubes. It is therefore expected that the cylinders be stiffer than the cubes.

The results will be discussed and analyzed in more depth later in this chapter.

Compression Testing: Modes of Failure

After reaching maximum load, some of the cubes and cylinders were loaded further to ultimate load. At ultimate load, the specimen fail completely producing macroscopic cracks and ruptures. For this thesis, it was desired to observe differences, if any, in the failure modes of the untreated and treated cubes and between the untreated and treated cylinders.

Cubes

Observing ultimate failure of the cubes was not very enlightening. Most of the untreated and treated cubes failed in splitting (Figure 4.5, type *a*). This is a result of the small specimen size. As the specimen was loaded, it bulged out laterally (horizontally). Vertical cracks formed perpendicular to this axis of maximum elongation. The cracks then propagated such that they intersected the bearing ends before shear (diagonal) cracks could form. One peculiar failure mode was observed with a treated cube: a "shell"

approximately 1/8"-1/4" (0.3-0.6 cm) thick spalled off the surface. Figure 7.5 illustrates this phenomenon. It is probable that a higher concentration of consolidant was near the surface. During loading, this surface zone of higher concentration acted as one consolidated unit. It is possible that the stronger, more consolidated surface zone sheared or delaminated away from the failing, weaker inner region of lower consolidant concentration.

Peculiar Failure of Treated Cube
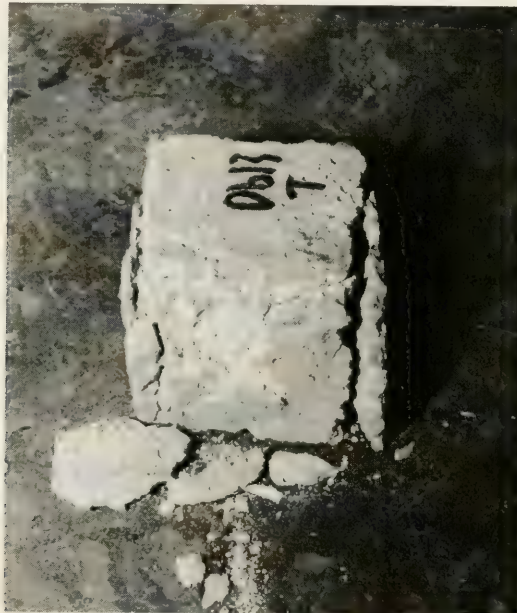
Figure 7.5

Most of the untreated and treated cubes failed by through-body splitting. This treated cube displayed an interesting mode of failure. A 1/8"-1/4" shell of material separated from the rest of the cube. This could be the result of a surface zone with higher consolidant concentration shearing or delaminating from a core of less concentrated consolidation.

143

<u>Cylinders</u>

In standard practice, compression tests are conducted on cylindrical specimen with height equal to twice the diameter so that surface rupture produced at failure will not intersect the bearing ends.[6] In theory, a uniformly stressed cylinder will break in the shape of a double cone with the vertex in the center of the cylinder. As depicted in Figure 4.5 (type b), this is called the "cone" failure mechanism. Treated cylinder Dc4, illustrated in Figure 7.6, clearly displayed this classic failure mechanism. Some of the cylinders failed by cone or cone-and-split modes, but many failed in splitting. Figure 7.7 illustrates an untreated cylinder that failed in a combination of cone-and-split, cone-and-shear, and simple splitting modes. The picture also points out that a 1/4"-1/2" (0.6 -1.3 cm) "shell" of concrete split away from the core.

Figure 7.8 illustrates a treated cylinder that exhibited predominantly splitting failure. A shell of concrete separated from the core, which could be attributed to shearing of the 1"-deep consolidated zone. Figure 7.9 illustrates another treated cylinder with a separated shell. Jones reports that interlaminar stresses exist between adjacent layers of composite materials, and that high interlaminar stresses are the basis for one type of failure mechanism in composites.[7] This explanation is inconclusive for the composite cylinder specimen, however, since several untreated cylinders displayed similar failures.

---

[6] Kenneth N. Derucher, George P. Korfiatis, and A. Samer Ezeldin, *Materials for Highway and Civil Engineers,* 3rd ed. (Englewood Cliffs, NJ: Prentice Hall, 1994), 104.

[7] Robert M. Jones, *Mechanics of Composite Materials* (Philadelphia, PA: Taylor & Francis, Inc., 1999), 260-261.
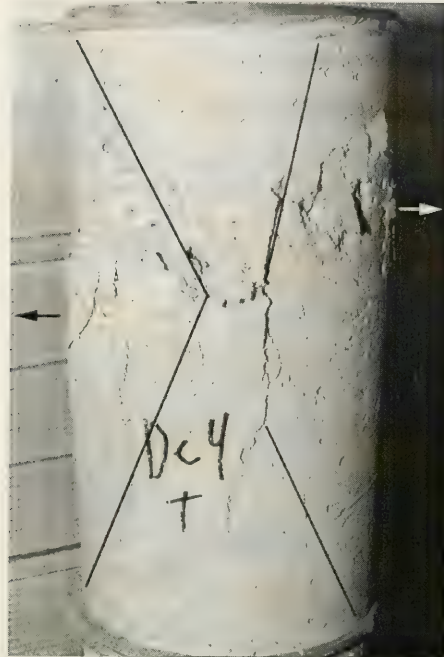
Figure 7.6

The lines superimposed on the photograph illustrate the double cone formed by the crack pattern. The arrows illustrate the lateral bulging of the cylinder at approximate mid-height.
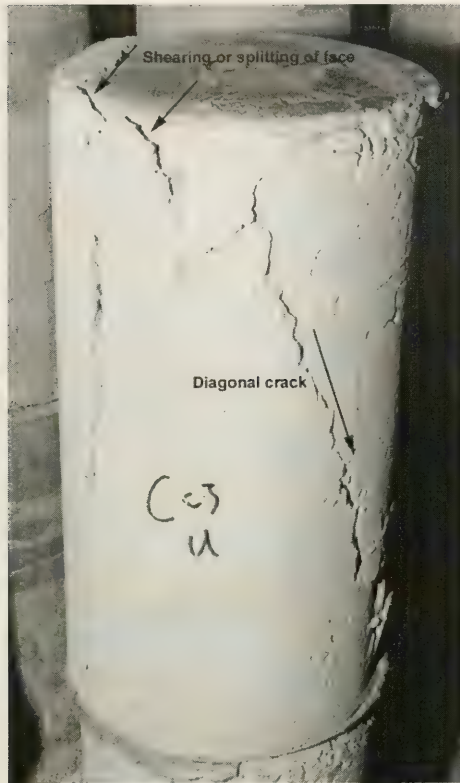
Figure 7.7

The diagonal crack on this untreated cylinder is typical of cone-and-split or cone-and-shear. The vertical crack on the left indicates simple split failure or columnar failure. A thin shell of concrete separated from the core even though this cylinder was untreated.

Figure 7.8

This treated cylinder exhibited predominantly splitting failure. A thin shell of concrete split away from the core. This could be due to the consolidant, but this theory is inconclusive since many untreated cylinders failed in a similar manner.

Failure of Treated Cylinder Fc1
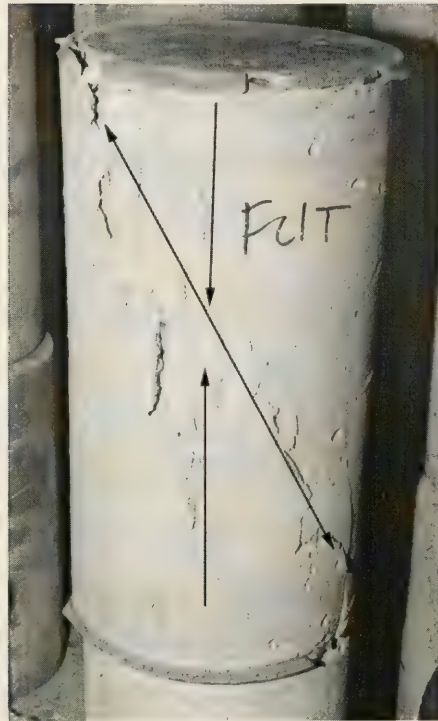
Figure 7.9

This cylinder exhibited splitting and cone modes of failure, illustrated by the superimposed arrows. In the top left of the cylinder, a thin shell has separated from the core. This could be due to the consolidant, but this theory is inconclusive since many untreated cylinders failed in a similar manner.

Discussion and Analysis of Testing

The testing program showed that fully (95%) penetrated 2" (5 cm) concrete cubes can sustain almost 13% more compressive load than untreated concrete cubes. Testing also showed that 6" (15.2 cm) diameter concrete cylinders consolidated to a depth of approximately 1" (2.54 cm) can sustain almost 6% more compressive load than untreated concrete cylinders. Even partial consolidation adds resistance to imposed load. Greater resistance to imposed load increases the material's allowable stress.

In any material, consolidation binds together the constituent grains of the substrate, which gives it greater resistance to imposed load. This mechanism could apply to concrete as well. Concrete is a peculiar material, however, because of the hydrating reactions during curing. Although treatment of other porous materials (stone, brick, etc.) should be done when the substrate is dry, concrete may never be fully "dry." Hydration reactions may continue for many years. During this testing program, the concrete was treated after only seven days of curing. The application of the treatment could have either hindered or improved hydration of the concrete. On one hand, the consolidant could have effectively sealed the inner core of concrete (especially in the cylinders) so that water was sealed in the concrete, improving hydration and increasing early strength. On the other hand, this sealing effect could hinder carbonation of the concrete and decrease early strength, since alkalis cannot be exchanged between the interior of the concrete and the environment. Thus, the fact that the treated concrete sustained a higher load may not be attributable solely to the consolidation mechanism (film forming or pore filling), but also to the consolidant's entrapment of water.

149

The testing program also showed that fully (95%) penetrated 2" (5 cm) concrete cubes have a 33% higher modulus of elasticity (greater stiffness) than untreated concrete cubes. However, testing showed that 6" (15.2 cm) diameter concrete cylinders consolidated to a depth of approximately 1" (2.54 cm) do not have a significantly higher modulus of elasticity than untreated concrete cylinders. When the specimen is uniformly loaded on over the untreated and treated portions of a composite specimen, the "average" modulus of elasticity over the entire section of the specimen is not much greater than that for the untreated cylinders.

In the cylinders, the 1" penetration depth of the consolidant over the 12" height of the cylinder accounts for 56% of the volume of the cylinder. If the modulus of elasticity of the inner untreated core is 2210 ksi (that of the untreated cylinder) and the modulus of elasticity of the treated shell is 2939 ksi (33% higher than the untreated cylinder, as observed in testing the cubes), one can apply the "rule of mixtures" presented in Chapter Four:

$$E_{composite} = E_{untreated}V_{untreated} + E_{treated}V_{treated}$$
$$E_{composite} = (2210 \text{ ksi})(0.44) + (2939 \text{ ksi})(.56) = 2618 \text{ ksi.}$$

The actual average E of the composite cylinders was 2352 ksi, less than that predicted by the rule. This predicted value is significantly higher than that recorded in actual testing, and it is significantly higher than the value of E of the untreated cylinder. The reality of composite action may not always coincide with theories presented in textbooks

The observed increases in sustained load and stress in the testing program were basically as expected. It was expected, however, that the modulus of elasticity for the treated cylinder specimen would have been higher than the testing showed. The recorded magnitudes of the loads and modulii of elasticity of the untreated and treated specimen were not drastically different, only differing by percentage points rather than by orders of magnitude. Detrimental stiffening was not observed in the cubes or cylinders.

The testing results alone, however, may not totally disprove the theory that composite and interface action in partially impregnated samples is detrimental to the performance of the element. The magnitude of the shear developed by transfer of stress due to loading and stiffening was not recorded or analyzed as part of this project. Only visual observations were used to detect the presence of shear forces. Because both the untreated and treated cylinders exhibited shearing of a thin shell, visual observations were inconclusive. However, the transfer of stress and the resulting shear development may be more critical a parameter in composite action of partially-impregnated substrates than the current test results and analysis could show.

CHAPTER EIGHT: CONCLUSIONS AND RECOMMENDATIONS

Conclusions

In most cases, consolidants are applied to deteriorated porous materials, particularly natural stone, with the intent of reintegrating sugaring or flaking grains. Although surface consolidants like Conservare OH are not usually applied with the intent of strengthening their substrates, they can also quite effectively strengthen deteriorated or undeteriorated material, as shown in work by several authors and in the testing program of this thesis. Particularly in materials that are fully impregnated with consolidant, material stiffening (increasing modulus of elasticity) is another effect of consolidation.

Numerous studies have shown that consolidants can effectively conserve and even strengthen building materials. In their published work, conservators often *qualitatively* state that over-strengthening and stiffening of consolidated substrates is undesirable. This thesis is an interdisciplinary approach to the study and testing of a consolidant, and it aimed to look critically and *quantitatively* at the effects of applying a consolidant to porous building materials. Theories and equations drawn from material mechanics and structural engineering quantitatively illustrated consolidating effects in regard to structural performance of the substrate. An increase in strength of the material after a fully-impregnating consolidation treatment allows increased load to be applied to the structural element. Stiffening makes the material more brittle, which could lead to increased cracking but does not increase the structural capacity of the element. Researchers are currently developing consolidating polymers with flexible linear

segments that make them more elastic and ductile. One elastified ethyl silicate has been shown to give the same flexural strength as a regular ethyl silicate, but with half the modulus of elasticity.[1] Such elastified polymers may reduce brittle cracking of treated materials, enhancing durability of the treated material and prolonging the service life of the element.

Almost all conservators agree that consolidants must penetrate at least through the undeteriorated zone of the substrate in order to minimize future surface loss. The conservators usually cite "locking" the treated zone to the undeteriorated zone as the reason for this specified penetration depth. Inadequate partial-depth penetration of consolidants into a load-bearing material can be, in fact, detrimental to the structural element itself and to the building as a whole. Partial-depth penetration of consolidant makes the substrate a two-part or three-part composite. In this thesis, the effect of composite action was examined for a uniaxially and uniformly loaded column. In the case of partial penetration of the cross-section with full penetration of the deteriorated zone, the column's treated shell carries more load than its untreated core. Requiring the treated shell to carry more load can cause failure in the shell if the compressive strength of the material is low before and after treatment. The most dangerous partial-penetration cases are those when material deteriorates behind a shallow treated zone that encases an inner band of untreated deteriorated material and/or a core that was not deteriorated before treatment. If deterioration continues such that the inner band and/or the core no

---

[1] Wendler discusses elastified ethyl silicates in detail in his article "Materials and Approaches for the Conservation of Stone."

longer contribute to the structural capacity of the element, the treated shell must carry the entire load applied to the column. If the treated material is weak such that its carrying capacity is exceeded by the applied stress, the treated shell will fail. This case is particularly alarming, since this "invisible" deterioration is difficult to monitor visually and will be manifest only after the shell begins to spall. Alternatively, failure can occur suddenly without warning, especially if applied stress or stress distributions shift rapidly.

It is important to reiterate that the example cases studied in Chapter Five imply a change in load (stress state) of the consolidated element. It is only when the stress state changes (i.e., when load is increased or decreased) after treatment that these situations come into play. As discussed, change in the magnitude and type of load and resulting stress state is a very real possibility for historic buildings.

Recommendations for Further Testing and Research

The case study and testing program presented in this thesis isolated one type of structural element (a column) and one type of load (uniform uniaxial). This thesis, then, should serve as a model for further testing and research on the mechanical properties of porous building materials. Deteriorated and undeteriorated, and unweathered and weathered specimens should be fully and partially impregnated with consolidant. Not only compression tests, but also tensile and bending tests should be carried out in larger-scale studies. Thermal and moisture cycling of the specimen should also be completed. Examining axial, bending, and shear stress distributions in the pure and composite materials will provide information applicable to a wider range of structural elements, not

just the column example in this thesis. Furthermore, combination loading (such as thermal + bending, compression + bending, etc.) should be completed, as this more closely depicts actual loading of a structural element.

As computer programs become more accessible to architects, conservators, and engineers, treated building materials can be modeled and virtually loaded and cycled. A finite element analysis program with sophisticated graphical output offers a view on the inner workings of the composite action and illustrates the stress distributions and deformations set up by the composite action through each zone of the specimen and at interfaces between zones. Such analysis provides detailed information that can guide the treatment specifications of the conservation project.

After testing, modeling and analysis, the conservation team must decide if consolidation is truly an appropriate treatment. Treatment should not be detrimental to the physical, chemical, mechanical, or structural properties of the building material. This thesis has shown that in some cases treatment can benefit the mechanical behavior and structural performance in some treatment scenarios. If treatment is deemed appropriate for the material *now*, the team must also consider *future* deterioration of the material and/or treatment. This thesis called attention to scenarios involving post-treatment deterioration that can cause failure of the building material. Many researchers are currently investigating the retreatability of conservation treatments in the event they fail or are superceded by better products. When treated materials are retreated, multi-part

composite materials are created. The performance of such complicated composite materials remains to be seen, and should be investigated in future research work.

APPENDICES

APPENDIX A: CONCRETE MATERIALS CHARACTERIZATION

Specific Gravity and Absorption of Coarse Aggregates

Objective

Determine the specific gravity and absorption of coarse aggregate. The specific gravity may be expressed as bulk specific gravity (BSG), bulk specific gravity saturated surface dry (BSG_{SSD}), or apparent specific gravity (BSG_{App}). BSG_{SSD} and absorption are based on aggregate after 24 hours soaking in water.

Standard

ASTM C 127-88 (Reapproved 1993): Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate.

Significance and Use

Determination of bulk specific gravity aids in calculating the volume occupied by the aggregate in concrete. BSG_{SSD} is used when the aggregate is wet; that is, if absorption has been satisfied. BSG is used when the aggregate is dry or is assumed dry. BSG_{App} pertains to the relative density of the solid making up the particles not including the pore space within the particles. Absorption is used to calculate the change in weight of an aggregate due to water absorbed in the pore spaces within the particles, compared to the dry condition.

Test Location

Systems Engineering Materials Lab
Room 11, Towne Building
University of Pennsylvania

Equipment Used

Balance:	U.S. units (lbs)
Weighing bucket:	6" diameter cylinder mold as is, and modified with 1/8" holes for drainage
Supplier:	American Paper Products
	1475 Park Avenue
	Alpha, NJ 08865
	908-454-9431
Water tank:	Large capacity tank

Paper towels for drying to SSD condition

Specimen

Crushed gravel
Supplier: George F. Kempf Building Materials Supply Co.
5800 Lindberg Boulevard
Philadelphia, PA 19143
1-800-326-5367

Special Conditions

As allowed in ASTM C 127 Section 8.2, the coarse aggregate was not dried prior to immersion, but was instead weighed in the natural state upon delivery.

The aggregate was not sieved prior to testing or use in the concrete mix; i.e., the entire bulk as delivered was included in this test.

Raw Data Obtained

Dry and SSD measurements in air

Measurement (lb)	Equation Variable	Trial 1	Trial 2	Trial 3
weight of dry bucket		0.72	0.70	0.72
dry bucket + gravel		4.91	5.61	4.75
dry mass gravel alone	A	4.19	4.91	4.03
bucket + SSD gravel		4.96	5.68	4.87
SSD gravel alone	B	4.24	4.98	4.15

Hydrostatic measurements

Measurement (lb)	Equation Variable	Trial 1	Trial 2	Trial 3
weight cylinder + chain in water		0.0	0.0	0.0
weight assembly + S SSD gravel in water	C	3.08	3.08	2.54

Calculations

Variables: A = weight of dry test sample in air
B = weight of SSD sample in air
C = weight of SSD sample in water

Equations: $BSG = \dfrac{A}{B-C}$ $BSG_{SSD} = \dfrac{B}{B-C}$ $Absorption = \dfrac{B-A}{A}$

Values:

Value	Trial 1	Trial 2	Trial 3	For Design
BSG	3.61	2.58	2.50	2.58
BSG$_{SSD}$	3.66	2.62	2.57	2.62
Absorption	1.2%	1.4%	2.9%	1.4%

From the author's previous experience, the values for Trial 1 seemed high. Trial 2's values seemed more realistic. Trial 3 was done to confirm Trial 2's values. For design purposes, the values from Trial 2 were used.

Specific Gravity and Absorption of Fine Aggregate

Objective

Determine the specific gravity and absorption of fine aggregate. The specific gravity may be expressed as bulk specific gravity (BSG), bulk specific gravity saturated surface dry (BSG$_{SSD}$), or apparent specific gravity (BSG$_{App}$). BSG$_{SSD}$ and absorption are based on aggregate after 24 hours soaking in water.

Standard

ASTM C 128-97: Standard Test Method for Specific Gravity and Absorption of Fine Aggregate.

Determination of bulk specific gravity aids in calculating the volume occupied by the aggregate in concrete. BSG_{SSD} is used when the aggregate is wet; that is, if absorption has been satisfied. BSG is used when the aggregate is dry or is assumed dry. BSG_{App} pertains to the relative density of the solid making up the particles not including the pore space within the particles. Absorption is used to calculate the change in weight of an aggregate due to water absorbed in the pore spaces within the particles, compared to the dry condition.

Test Location

Architectural Conservation Lab
University of Pennsylvania

Equipment Used

Balance:	Denver Instrument XE-510 (digital, ±0.01g)
	OHAUS Dial-o-gram (analog, ±0.1g)
Flask:	1000 ml volumetric flask
Mold:	Cone mold per ASTM C 109
Non-absorbent plate:	Frosted glass plate
Tamper:	Tamper per ASTM C 109 (rectangular, not round)
Dryer:	Hair dryer to bring to SSD condition

Specimen

Concrete sand, passing No. 4 sieve

Supplier:	George F. Kempf Building Materials Supply Co.
	see address above

Special Conditions

As allowed in ASTM C 127 Section 8.2, the fine aggregate was not dried prior to immersion, but was instead weighed in the natural state upon delivery.

Raw Data Obtained

Measurement (g)	Equation Variable	Trial 1	Trial 2
container 1		13.96	13.79
container 1 + dry sand		287.83	260.69
sand alone	A	273.87	246.90
flask		258.37	257.28
flask + water to line	B	1255.0	1251.5
container 2		13.96	13.72
container 2 + SSD sand		308.66	279.56
SSD sand alone	S	294.7	265.84
SSD sand + flask + water	C	1413.8	1396.1

Calculations

Variables:
A = weight of dry test sample in air
B = weight of flask filled with water
S = weight of SSD sample
C = weight of flask + sand + water to line

Equations:
$$BSG = \frac{A}{B+S-C} \qquad BSG_{SSD} = \frac{S}{B+S-C} \qquad Absorption = \frac{S-A}{A}$$

Values:

Value	Trial 1	Trial 2	For Design
BSG	2.02	2.04	2.03
BSG$_{SSD}$	2.17	2.19	2.18
Absorption	7.6%	7.6%	7.6%

The average value between Trials 1 and 2 were used for the design.

Bulk Density of Coarse Aggregate

Objective

Determine the bulk density ("unit weight") of coarse aggregate.

Standard

ASTM C 29-97: Standard Test Method for Bulk Density ("Unit Weight") and Voids in Aggregate

Significance and Use

Determination of bulk density values necessary for selecting proportions for concrete mixtures.

Test Location

Systems Engineering Materials Lab

Equipment Used

Balance:	U.S. units (lbs)
Measure:	6" diameter cylinder mold
Supplier:	American Paper Products
Tamping Rod:	5/8" diameter, 24" long straight steel rod

Raw Data Obtained

Cylinder dimensions: inside diameter = 6"
inside height = 12"
wall thickness = 0.75"
volume = 339.3 in^3 = 0.196 ft^3

Calibration of measure

Measurement	Value
weight of bucket (lb)	0.72
weight of bucket + water (lb)	13.08
weight of water alone (lb)	12.36
temperature of water (°C)	16
density of water (lb/ft^3)*	62.36
Volume of measure $= \dfrac{\text{weight of water}}{\text{density of water}}$	0.198 ft^3

* from ASTM C 29 Table 3

The volume obtained from measuring the dimensions and from the density of water compare well.

161

Rodding procedure

Measurement	Trial 1	Trial 2
weight bucket + stone (lb)	22.04	21.99
weight of stone alone (lb)	21.32	21.27

Calculations

Value	Trial 1	Trial 2
bulk density $= \dfrac{\text{weight of stone}}{\text{volume of measure}}$	107.7 lb/ft^3	107.42 lb/ft^3

Single-operator deviation allowed by ASTM C 29 is 2.5 lb/ft^3. Thus, the average of the values for Trials 1 and 2 were used. Average bulk density of the coarse aggregate used in the design calculations is 107.6 lb/ft^3.

Moisture Content of Fine and Coarse Aggregates

Objective

Determine the percentage of evaporable moisture (moisture content) in a sample of aggregate by drying both the surface moisture and moisture in the pores of the aggregate. Water chemically combined with minerals is not included by this test.

Standard

ASTM C 566-97: Standard Test Method for Total Evaporable Moisture Content of Aggregate by Drying.

Significance and Use

Determination of the moisture content of the aggregates aids in adjusting the water added to a concrete mix. Excess moisture content means that less water should be added to the plastic concrete mix.

Test Location

Architectural Conservation Lab
University of Pennsylvania

Equipment Used

Balance: Denver Instrument XE-510 (digital, ±0.01g)
Source of heat: Temperature-regulated oven
Sample container: Aluminum pie pans

Coarse Aggregate

Raw Data Obtained

Oven temperature = 110°C.

Measurement (g)	Variable	Trial 1	Trial 2
mass of container		13.92	13.80
mass container + "wet" stone	W	295.11	361.75
mass container + dry stone – day 1		295.02	361.68
mass container + dry stone – day 2	D	295.03	361.65

162

Values for day 2 were used to calculate the moisture content.

Calculations

Variables: W = mass of original sample
D = mass of dried sample

Equations:

$$\text{Moisture content} = \frac{W - D}{D}$$

Values:

Value	Trial 1	Trial 2
Moisture content (%)	0.0	0.0

The moisture content for the coarse aggregate is negligible.

Fine Aggregate

Raw Data Obtained

Oven temperature = 110°C.

Measurement (g)	Variable	Trial 1	Trial 2
mass of container		14.50	13.8
mass container + "wet" stone	W	334.00	266.0
mass container + dry stone – day 1		321.21	256.30
mass container + dry stone – day 2	D	320.9	256.0

Values for day 2 were used to calculate the moisture content.

Calculations

Variables: W = mass of original sample
D = mass of dried sample

Equations:

$$\text{Moisture content} = \frac{W - D}{D}$$

Values:

Value	Trial 1	Trial 2
Moisture content (%)	4.3%	4.1%

ASTM C566 mandates that two properly conducted tests by the same operator I the same lab should not differ by more than 0.79% -- this condition has been met. The average moisture content of Trials 1 and 2 is 4.2%.

Sieve Analysis of Fine and Coarse Aggregates

Objective

Obtain particle size distribution of fine and coarse aggregates by sieving.

Standard

ASTM C 136-84a: Standard Method for Sieve Analysis of Fine and Coarse Aggregates

Significance and Use

Determination of the grading of materials to be used as aggregates. Results are used to characterize the aggregates used and to determine compliance of the material to written specifications, if they exist. In concrete mix design, the particle size distribution can determine workability of the plastic concrete.

Test Location

Architectural Conservation Lab
University of Pennsylvania

Equipment Used

Balance: Denver Instrument XE-510 (digital, ±0.01g)
Sieves: Standard sieves
Mechanical shaker: Combustion Engineering Sieve Shaker Model RX-86

Coarse Aggregate

Specimen

Thoroughly mix the aggregate and reduce it to the sample size as outlined in ASTM C 136 Section 6.4.

Special Conditions

Prior to sampling, the bulk was not sieved so as to be retained on the No. 4 sieve, nor was dust in the bulk removed. The coarse aggregate was tested and used in the concrete mix as it was delivered.

Raw Data Obtained

Sample obtained

Measurement (g)	Value
mass of container	13.95
mass container + dry sample	495.63
mass dry sample alone	481.68

Sieving results

Sieve	Mesh Size (mm)	Mass Container (g)	Mass container + sample (g)
No. 4	4.7	4.06	415.13
No. 8	2.38	3.87	70.94
No. 16	1.18	4.07	6.71
No. 30	0.6	4.02	4.08
No. 50	0.3	3.87	3.99
No. 100	0.15	3.99	4.06
No. 200	0.075	4.14	4.15
Pan	--	4.04	4.48

Calculations

Mass retained = (mass container + sample) – (mass container)

$$\% \text{ Retained} = \frac{\text{Mass retained}}{\text{Mass of dry sample}}$$

Cumulative % Retained = % Retained$_n$ + % Retained$_{n+1}$ + ... + % Retained$_{n+i}$
% Passing = % Retained$_n$ - % Retained$_{n+1}$

Sieve	Mass Retained (g)	% Retained	Cum. % Ret.	% Passing
No. 4	411.07	85.3	85.3	14.7
No. 8	67.07	13.9	99.3	0.7
No. 16	2.64	0.5	99.8	0.2
No. 30	0.06	0.0	99.8	0.2
No. 50	0.12	0.0	99.8	0.1
No. 100	0.07	0.0	99.9*	0.1
No. 200	0.01	0.0	99.9*	0.1
Pan	0.44	0.1	100.0	0.0
Σ	481.48	100.0		
Δ Mass (loss)	0.2	neglect		

* due to rounding

Fine Aggregate

Specimen

Thoroughly mix the aggregate and reduce it to the sample size as outlined in ASTM C 136 Section 6.3.

Special Conditions

Prior to sampling, the bulk was sieved through the No. 4 sieve.

Sample obtained

Measurement (g)	Value
mass of container	14.5
mass container + dry sample	563.70
mass dry sample alone	549.2

Sieving results (all material passed No. 4)

Sieve	Mesh Size (mm)	Mass Container (g)	Mass container + sample (g)
No. 8	2.38	4.09	26.24
No. 16	1.18	4.25	94.73
No. 30	0.6	3.68	165.60
No. 50	0.3	4.23	209.7
No. 100	0.15	3.72	73.07
No. 200	0.075	3.95	10.12
Pan	--	4.06	5.76

Calculations

Mass retained = (mass container + sample) − (mass container)

$$\% \text{ Retained} = \frac{\text{Mass retained}}{\text{Mass of dry sample}}$$

Cumulative % Retained = % Retained$_n$ + % Retained$_{n+1}$ + ... + % Retained$_{n+i}$

% Passing = % Retained$_n$ - % Retained$_{n+1}$

$$\text{Fineness Modulus} = \frac{\sum \% \text{ passing (No. 8 through No.100)}}{100}$$

Sieve	Mass Retained (g)	Correct Mass Ret. (g)*	% Retained	Cum. % Ret.	% Passing
No. 8	22.15	21.09	3.8	3.8	96.2
No. 16	90.48	89.42	16.3	20.1	79.9
No. 30	204.84	203.78	29.3	86.5	42.8
No. 50	161.92	160.86	37.1	57.2	13.5
No. 100	69.35	68.29	12.4	99.0	1.0
No. 200	6.17	5.11	0.9	99.9	0.1
Pan	1.7	0.64	0.1	100.0	0.0
$\sum$	556.61	549.2			
Δ Mass (gain)	7.41	0			

* subtract 7.41/7 to each sieve

Fineness Modulus = 2.67

166

Particle Size Distribution:
Fine Aggregate
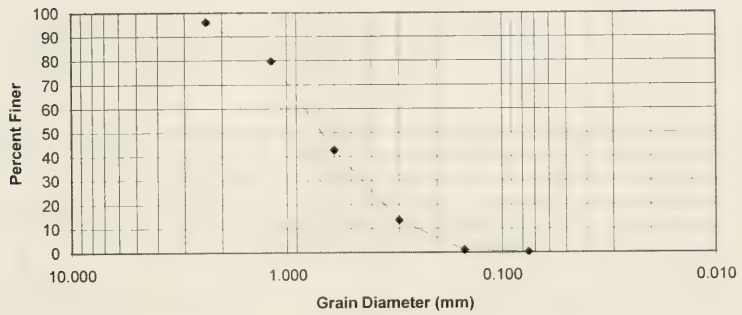
Visual Observation of Fine and Coarse Aggregate

Objective

    To describe and characterize the aggregates.

Standard

    No specific standard. Munsell Soil Color Charts were used to describe color. Sphericity and roundness were described using Figure A.5 in Clive Orten, et al., *Pottery in Archaeology* (Cambridge: Cambridge University Press, 1993), 239.

Observations

    Color observations made with the naked eye. Sphericity and roundess observed with a stereoscope.

Coarse Aggregate

    Bulk sample   *Color* 2.5Y 8/0 to 2.5Y 4/0 (white to dark gray)
                          Sphericity/Roundness low/very angular to sub-angular

Portion retained on sieve	Color	Sphericity	Roundness
No. 4	2.5Y 8/0 (white) to 2.5Y 4/0 (dark gray), and 2.5Y 7/4 (pale yellow)	low	angular to sub-rounded
No. 8	2.5Y 8/0 (white) to 2.5Y 4/0 (dark gray), 2.5Y6/6 (olive yellow)	low	sub-angular to rounded
No. 16	2.5Y 8/0 (white) to 2.5Y 4/0 (dark gray), 2.5Y6/6 (olive yellow)	low	sub-angular to rouded
No. 30 - Pan	2.5Y 8/0 (white) to 2.5Y 4/0 (dark gray),	powder	powder

Fine Aggregate

    Bulk sample   *Color* 10YR 6/8 (brownish yellow)
                          Sphericity/Roundness low/sub-rounded to rounded

Portion retained on sieve	Color	Sphericity	Roundness
No. 8	10YR 8/1 (white) to 10YR 8/8 (yellow)	low	sub-rounded
No. 16	entire 10YR range	high	sub-rounded
No. 30	entire 10YR range	low	sub-rounded
No. 50	10YR 7/6 (yellow)	high	sub-rounded
No. 100	10YR 6/6 (brownish yellow)	low	sub-rounded
No. 200	10YR 7/6 (yellow)	low	rounded
Pan	10YR 7/6 (yellow)	powder	powder

168

APPENDIX B: SPECIMEN PRODUCTION AND TREATMENT

Production and Treatment Schedule

Activity	Batch A	Batches B & C	Batches D & E	Batches F & G
Batch concrete	23 Nov 1999	13 Jan 2000	14 Jan 2000	15 Jan 2000
De-mold concrete	30 Nov 1999	14 Jan 2000	15 Jan 2000	16 Jan 2000
Polish cubes	5 Jan 2000	19 Jan 2000	19 Jan 2000	19 Jan 2000
Cap cylinders	--	18, 20 Jan 2000	18, 20 Jan 2000	18, 20 Jan 2000
Begin immersion (cubes)	12 Dec 1999	20 Jan 2000	21 Jan 2000	22 Jan 2000
Brush application (cylinders)	--	20 Jan 2000	21 Jan 2000	22 Jan 2000
Remove from immersion (cubes)	15-17 Dec 1999	25 Jan 2000	26 Jan 2000	27 Jan 2000
Cut sections in cubes	5 Jan 2000	1 Feb 2000	1 Feb 2000	1 Feb 2000
Take cores from cylinders	--	1 Feb 2000	1 Feb 2000	1 Feb 2000
Microdrop absorption test	9 Jan 2000	4-10 Feb 2000	4-10 Feb 2000	4-10 Feb 2000
21-day cure finished	--	15 Feb 2000	16 Feb 2000	17 Feb 2000
Compression testing	17 Jan 2000	22 Feb 2000	22 Feb 2000	22 Feb 2000
Total days from treatment end to compression test	33 days	28 days	27 days	26 days
Total days from batch to compression test	55 days	40 days	39 days	38 days

169

Specimen Use

Specimen Nomenclature: first letter = batch (A-G)
                         second letter: specimen type (b=cube, c=cylinder)

Batch A

Specimen	Batched	Treatment	Date Treated	Testing Use
Ab1	23 Nov 1999	untreated	--	preliminary compression testing
Ab2	23 Nov 1999	untreated	--	preliminary compression testing
Ab3	23 Nov 1999	untreated	--	preliminary compression testing
Ab4	23 Nov 1999	untreated	--	preliminary compression testing
Ab5	23 Nov 1999	untreated	--	not used
Ab6	23 Nov 1999	untreated	--	preliminary microdrop absorption
Ab7	23 Nov 1999	5-day immersion	12-17 Dec 1999	preliminary compression testing
Ab8	23 Nov 1999	5-day immersion	12-17 Dec 1999	preliminary compression testing
Ab9	23 Nov 1999	5-day immersion	12-17 Dec 1999	preliminary compression testing
Ab10	23 Nov 1999	4-day immersion	12-16 Dec 1999	preliminary microdrop absorption
Ab11	23 Nov 1999	3-day immersion	12-15 Dec 1999	preliminary microdrop absorption
Ab12	23 Nov 1999	untreated	--	preliminary compression testing
Ab13	23 Nov 1999	5-day immersion	12-17 Dec 1999	preliminary microdrop absorption
Ab14	23 Nov 1999	untreated	--	not used
Ab15	23 Nov 1999	destroyed at de-molding	--	--
Ac1	23 Nov 1999	brush application	8 Jan 2000 (3x3) 9 Jan 2000 (3x4)	treatment application trial
Ac2	23 Nov 1999	brush application	8 Jan 2000 (3x3) 9 Jan 2000 (3x5)	treatment application trial
Ac3	23 Nov 1999	untreated	--	not used
Ac4	23 Nov 1999	untreated	--	not used
Ac5	23 Nov 1999	untreated	--	not used.

Batch B

Specimen	Batched	Treatment	Date Treated	Testing Use
Bb1	13 Jan 2000	5-day immersion	21-26 Jan 2000	microdrop absorption
Bb2	13 Jan 2000	untreated	--	not used
Bb3	13 Jan 2000	untreated	--	compression
Bb4	13 Jan 2000	untreated	--	compression
Bb5	13 Jan 2000	5-day immersion	21-26 Jan 2000	microdrop absorption
Bb6	13 Jan 2000	untreated	--	compression
Bb7	13 Jan 2000	5-day immersion	20-25 Jan 2000	compression
Bb8	13 Jan 2000	5-day immersion	20-25 Jan 2000	compression
Bb9	13 Jan 2000	untreated	--	not used
Bb10	13 Jan 2000	untreated	--	not used
Bb11	13 Jan 2000	untreated	--	compression
Bb12	13 Jan 2000	5-day immersion	20-25 Jan 2000	compression
Bb13	13 Jan 2000	untreated	--	compression
Bb14	13 Jan 2000	untreated	--	not used
Bb15	13 Jan 2000	5-day immersion	21-26 Jan 2000	microdrop absorption
Bc1	13 Jan 2000	untreated	--	compression
Bc2	13 Jan 2000	untreated	--	compression
Bc3	13 Jan 2000	brush application	20 Jan 2000	compression
Bc4	13 Jan 2000	brush application	20 Jan 2000	compression
Bc5	13 Jan 2000	untreated	--	compression

Batch C

Specimen	Batched	Treatment	Date Treated	Testing Use
Cc1	13 Jan 2000	brush application	20 Jan 2000	compression
Cc2	13 Jan 2000	*not capped*	--	not used
Cc3	13 Jan 2000	untreated	--	compression
Cc4	13 Jan 2000	untreated	--	compression
Cc5	13 Jan 2000	brush application	20 Jan 2000	compression
Ccx	13 Jan 2000	*not capped,* brush application	20 Jan 2000	microdrop absorption

171

Batch D

Specimen	Batched	Treatment	Date Treated	Testing Use
Db1	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db2	14 Jan 2000	untreated	–	microdrop absorption
Db3	14 Jan 2000	untreated	--	compression
Db4	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db5	14 Jan 2000	untreated	--	compression
Db6	14 Jan 2000	untreated	--	compression
Db7	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db8	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db9	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db10	14 Jan 2000	untreated	--	compression
Db11	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db12	14 Jan 2000	untreated	--	not used
Db13	14 Jan 2000	5-day immersion	21-26 Jan 2000	compression
Db14	14 Jan 2000	untreated	--	compression
Db15	14 Jan 2000	untreated	--	not used
Dc1	14 Jan 2000	untreated	--	compression
Dc2	14 Jan 2000	*not capped*	--	not used
Dc3	14 Jan 2000	brush application	21 Jan 2000	compression
Dc4	14 Jan 2000	brush application	21 Jan 2000	compression
Dc5	14 Jan 2000	brush application	21 Jan 2000	compression

Batch E

Specimen	Batched	Treatment	Date Treated	Testing Use
Ec1	14 Jan 2000	untreated	--	compression
Ec2	14 Jan 2000	brush application	21 Jan 2000	compression
Ec3	14 Jan 2000	brush application	21 Jan 2000	compression
Ec4	14 Jan 2000	untreated	--	compression
Ec5	14 Jan 2000	untreated	--	compression
Ecx	14 Jan 2000	*not capped*, untreated	--	microdrop absorption

172

Batch F

Specimen	Batched	Treatment	Date Treated	Testing Use
Fb1	15 Jan 2000	untreated	--	compression
Fb2	15 Jan 2000	untreated	--	compression
Fb3	15 Jan 2000	5-day immersion	22-27 Jan 2000	compression
Fb4	15 Jan 2000	untreated	--	compression
Fb5	15 Jan 2000	untreated	--	compression
Fb6	15 Jan 2000	untreated	--	compression
Fb7	15 Jan 2000	5-day immersion	22-27 Jan 2000	compression
Fb8	15 Jan 2000	untreated	--	not used
Fb9	15 Jan 2000	untreated	--	compression
Fb10	15 Jan 2000	5-day immersion	22-27 Jan 2000	compression
Fb11	15 Jan 2000	1-hour immersion	22 Jan 2000	microdrop absorption
Fb12	15 Jan 2000	1-hour immersion	22 Jan 2000	microdrop absorption
Fb13	15 Jan 2000	5-day immersion	22-27 Jan 2000	compression
Fb14	15 Jan 2000	5-day immersion	22-27 Jan 2000	compression
Fb15	15 Jan 2000	untreated	--	microdrop absorption
Fc1	15 Jan 2000	brush application	22 Jan 2000	compression
Fc2	15 Jan 2000	untreated	--	compression
Fc3	15 Jan 2000	*not capped*	--	not used
Fc4	15 Jan 2000	brush application	22 Jan 2000	compression
Fc5	15 Jan 2000	untreated	--	compression

Batch G

Specimen	Batched	Treatment	Date Treated	Testing Use
Gc1	15 Jan 2000	brush application	22 Jan 2000	compression
Gc2	15 Jan 2000	untreated	--	compression
Gc3	15 Jan 2000	brush application	--	compression
Gc4	15 Jan 2000	untreated	--	compression
Gc5	15 Jan 2000	brush application	22 Jan 2000	compression
Gcx	15 Jan 2000	*not capped,* untreated	--	not used

173

APPENDIX C: MICRODROP ABSORPTION TESTING

Statistics Used in Microdrop Absorption Analysis

Average
$$\bar{x} = \frac{\sum x_i}{N_i}$$

Standard Deviation
$$s = \sqrt{\frac{N_i \sum x_i - (\sum x_i)^2}{N_i(N_i - 1)}}$$

Variance
$$variance = s^2$$

Confidence Limit
$$c = \pm \frac{ts}{\sqrt{N}}$$, t is Student's t found in tables as a function of given confidence level and degrees of freedom

Confidence Interval
$$\mu = \bar{x} \pm c$$

Sum of Squares
$$S_1 = \sum(x_{i1} - \bar{x}_1)^2 \ , \ \ S_2 = \sum(x_{i2} - \bar{x}_2)^2,$$ etc. for each treatment type

Within Treatment Sum of Squares
$$S_R = S_1 + S_2 + ... + S_k,$$ where k = number of treatments

Within Treatment Mean Square
$$s_R^2 = \frac{S_R}{\upsilon_R},$$ where v_R = degrees of freedom = N - k

Between Treatment Sum of Squares
$$S_T = N_1(\bar{x}_1 - \bar{\bar{x}})^2 + ... + N_k(\bar{x}_k - \bar{\bar{x}}),$$ where $\bar{\bar{x}}$ is the grand average of the pooled (all treatments) data

Between Treatment Mean Square
$$s_i^2 = \frac{S_T}{\upsilon_T},$$ where v_T = degrees of freedom = k-1

Ratio of Mean Squares
$$r = \frac{s_T^2}{s_R^2}$$

F-Test
The F-test allows estimation of whether a significant difference exists in the precision of two sets of data. The ratio of the variances (s_1^2/s_2^2, where s_1^2 is the larger absolute variance) is compare to critical values of F listed in tables as a function of the degrees of freedom of both samples at a given confidence level. If $s_1^2/s_2^2 > F$, there is a statistically significant difference between the two samples.

Determination of Microdrop Size and Volume

Objective
> Determine the size and volume of a drop of water to be used in the microdrop absorption test.

Standard
> RILEM Group 25 P.E.M. "Water Drop Absorption, Test No. II.8a," and "Water Drop Absorption, Test No. II.8b." *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978; A.P. Ferreiro Pinto, *Conservação de Pedras Graniticas. Estudo da Acção de Hidrófugos.* ITG22, Labóratorio Nacional de Engenharia Civil, Lisbon, Portugal.

Significance and Use
> The size of a microdrop of water determines how closely spaced microdrops of water can be placed on a surface undergoing the microdrop absorption test. The volume of the microdrop of water determines the height above the surface the water is to be dropped in the microdrop absorption test.

Test Location
> Architectural Conservation Lab
> University of Pennsylvania

Equipment Used
> Balance: Denver Instrument XE-510 (digital, ±0.01g)
> Pipettes: PCR pipettes (graduated every 1μl, 10μl capacity)
>   Supplier: Drummond Scientific, Broomall, PA
> Digital stopwatch
> Small glass beaker
> Deionized water

Procedure
1. Weigh dry, empty glass beaker.
2. Fill pipette to 10μl gradation.
3. Drop water into glass beaker.
4. Repeat for a total of 20 drops.
5. Weigh beaker + 20 drops.
6. Calculate volume of drops by dividing by the density of water.

Raw Data Obtained

Trial	Beaker Weight (g)	Beaker + 20 drops (g)	20 drops alone (g)
1	30.32	30.51	0.19
2	30.33	30.53	0.20
3	30.32	30.53	0.21

Calculations

Number of trials: $N = 3$

Average weight of 20 drops: $\bar{x}_{20drops} = 0.20$ g

Standard deviation of weight of 20 drops in 3 trials: $s = 0.01$ g

Confidence Level	t	Confidence interval, 20 drops	Confidence interval, 1 drop
90%	2.920	0.20 ± 0.02 g	0.01 ± 0.001 g
95%	4.303	0.20 ± 0.02 g	0.01 ± 0.001 g
99%	9.925	0.20 ± 0.06 g	0.01 ± 0.003 g

Temperature of water = 68°F (20°C)
Density of water: $\rho_{68°F}$ = 0.99821 g/cm^3

Volume of microdrop (90% confidence interval): 0.01 ± 0.001 mL, or 10 ± 0.1 µl

Volume of drop	Drop height above specimen
1 ml	1 cm
0.1 ml	0.5 cm
10 µl	1 cm
4 µl	1 cm

The diameter of the drop on the concrete cross-section was approximately 0.25" (0.64 cm), determined visually.

Microdrop Absorption Test: Procedure

Objective

Determine the absorption time of a microdrop of water on a cross-section of untreated and treated substrates. Compare the absorption times to determine if there is a statistically difference between the two, indicating that the treatment is present at the location of the water drop.

Standard

RILEM Group 25 P.E.M. "Water Drop Absorption, Test No. II.8a," and "Water Drop Absorption, Test No. II.8b." *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978; A.P. Ferreiro Pinto, *Conservação de Pedras Graniticas. Estudo da Acção de Hidrófugos.* ITG22, Labóratorio Nacional de Engenharia Civil, Lisbon, Portugal.

Significance and Use

Short-term water-repellency of ethyl silicate consolidants (like Conservare OH) during polymerization allows the comparison in water drop absorption times on treated and untreated substrates. This method relies on statistically analysis to determine the significance of the variance between the absorption times on the different substrates.

Test Location

Architectural Conservation Lab, University of Pennsylvania

Equipment Used

Balance: Denver Instrument XE-510 (digital, ±0.01g)

176

Pipettes: PCR pipettes (graduated every 1μl, 10μl capacity)
                   Supplier: Drummond Scientific, Broomall, PA
Digital stopwatch
Frosted glass
Deionized water
Temperature-controlled oven
Dessicator chamber

Specimen

2" (5 cm) concrete cubes, untreated and treated with Conservare OH, andcut in half to reveal internal cross-section

0.875" (2.2 cm) diameter by 1.875" (4.6 cm) long cores drilled from untreated and treated concrete cylinders

Procedure

1. Dry specimen in 60°C oven to constant mass. Cool prior to testing in dessicator chamber.
2. Place several microdrops on frosted glass to determine time for evaporation.
3. From appropriate height above specimen surface, drop microdrop onto specimen.
4. Determine time of absorption. "Absorption" is indicated by the flattening of the water drop such that no reflective water film remained on the surface when viewed from a 30-45° angle from the surface. "Absorption" does mandate a dry surface.
5. Repeat for several trials, drying and cooling the specimen as before.
6. Calculate statistics for the untreated and treated specimen. Apply the F-test to determine if a significant difference exists in the precision of the two sets of treatments. Carry out analysis of variance between the two sets of treatments.

Note: Evaporation time of drops on glass surface was so long (40 minutes), it was deemed insignificant.

Preliminary Microdrop Absorption Test
Batch A Cubes

Untreated Specimen ("a" and "b" indicate the two halves produced when one 2" cube is cut in half)

Microdrop Absorption Time (minutes)						
Specimen		0"	1/2"	Center	1-1/2"	2"
Ab6a	Axis 1	0.84	0.75	1.85	0.51	0.76
	Axis 2	0.91	1.39	1.84	0.95	1.36
Ab6b	Axis 1	1.15	0.00	2.63	1.51	0.59
	Axis 2	1.22	1.64	1.82	1.90	0.65

177

Statistical Analysis					
Statistic	**0"**	**1/2"**	**Center**	**1-1/2"**	**2"**
Minimum Value	0.84	0.00	1.82	0.51	0.59
Maximum Value	1.22	1.64	2.63	1.90	1.36
Average Value	1.03	0.95	2.04	1.22	0.84
Standard Dev.	0.18	0.73	0.40	0.61	0.35
Variance	0.03	0.54	0.16	0.37	0.13
Conf. Limit, 90%	±0.22	±0.86	±0.47	±0.72	±0.42
Conf. Limit, 95%	±0.29	±1.17	±0.63	±0.97	±0.56
Sum of Squares	0.10	1.61	0.47	1.12	0.38

F-test Matrix					
s_1^2/s_2^2	**0"**	**1/2"**	**Center**	**1-1/2"**	**2"**
0"	n/a	15.96	4.68	11.12	3.72
1/2"	15.96	n/a	3.41	1.43	4.29
Center	4.68	3.41	n/a	2.38	1.26
1-1/2"	11.12	1.43	2.38	n/a	2.99
2"	3.72	4.29	1.26	2.99	n/a

Critical F (N=4, 95% confidence) = 9.28. Those values highlighted are greater than Critical F, meaning there is a statistically significant difference between those positions on the untreated specimen. This difference could represent concrete heterogeneity or experimental error. Because these specimen are untreated, pooling data of from all positions is justified.

It is unknown why the center position has such a higher absorption time that the other positions, but there could microcracks due to shrinkage at the edges. The scatter in absorption time between the axes and the two halves could be due to porosity variations across the cross-section.

Preliminary Microdrop Test:
Untreated Cubes

Position on Cross-Section
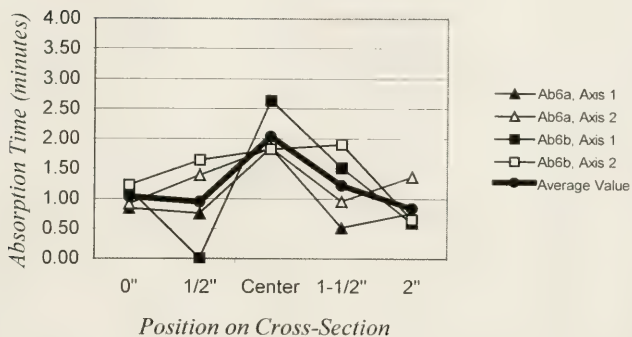
Pooled Data: Preliminary Untreated Cubes	
Statistic	**Value**
Minimum Value	0.00
Maximum Value	2.63
Average Value	1.21
Standard Dev.	0.62
Variance	0.39
Conf. Limit, 90%	±0.24
Conf. Limit, 95%	±0.29
Sum of Squares	7.37
Within Treatment Mean Sq.	0.39

3-day Treated Specimen

Microdrop Absorption Time (minutes)						
Specimen		0"	1/2"	Center	1-1/2"	2"
Ab11a	Axis 1	1.70	1.46	2.86	1.88	1.52
	Axis 2	1.76	1.82	1.33	0.88	1.61
Ab11b	Axis 1	2.33	1.26	2.95	1.64	3.32
	Axis 2	1.08	3.58	1.12	2.62	1.71

Statistical Analysis					
Statistic	0"	1/2"	Center	1-1/2"	2"
Minimum Value	1.08	1.26	1.12	0.88	1.52
Maximum Value	2.33	3.58	2.95	2.62	3.32
Average Value	1.72	2.03	2.07	1.76	2.04
Standard Dev.	0.51	1.06	0.97	0.72	0.86
Variance	0.26	1.12	0.95	0.51	0.73
Conf. Limit, 90%	±0.60	±1.24	±1.14	±0.84	±1.01
Conf. Limit, 95%	±0.81	±1.68	±1.55	±1.14	±1.36
Sum of Squares	0.78	3.36	2.85	1.54	2.20

F-test Matrix					
s_1^2/s_2^2	0"	1/2"	Center	1-1/2"	2"
0"	n/a	4.29	3.63	1.97	2.81
1/2"	4.29	n/a	1.18	2.18	1.53
Center	3.63	1.18	n/a	1.85	1.29
1-1/2"	1.97	2.18	1.85	n/a	1.43
2"	2.81	1.53	1.29	1.43	n/a

Critical F (N=4, 95%) = 9.28. All values are less than Critical F, so there is no statistically significant difference between the positions of the 3-day treated specimen. The data from all positions can be pooled.

There is much scatter in the absorption times at each position. However, the average absorption times are higher than those for the untreated specimen. This indicates a "homogenizing effect" and hydrophobizing caused by the Conservare OH treatment.

Preliminary Microdrop Test:
Cubes Immersed 3 Days

Absorption Time (minutes) vs *Position on Cross-Section*

Legend:
- Ab11a, Axis 1
- Ab11a, Axis 2
- Ab11a, Axis 1
- Ab11b, Axis 2
- Average Value
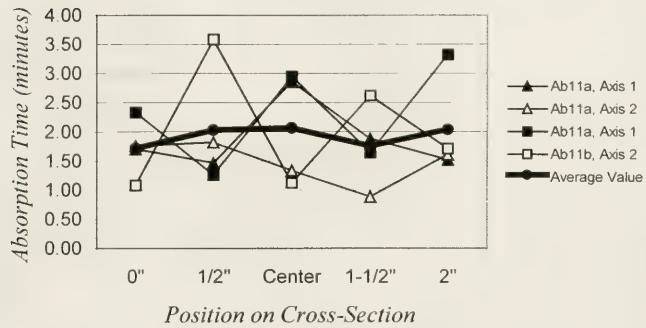
Pooled Data: Preliminary 3-day Treated Cubes	
Statistic	**Value**
Minimum Value	0.88
Maximum Value	3.58
Average Value	1.92
Standard Dev.	0.77
Variance	0.59
Conf. Limit, 90%	±0.30
Conf. Limit, 95%	±0.36
Sum of Squares	11.20
Within Treatment Mean Sq.	0.59

4-day Treated Specimen

Microdrop Absorption Time (minutes)						
Specimen		0"	1/2"	Center	1-1/2"	2"
Ab10a	Axis 1	1.49	0.67	0.68	1.18	1.92
	Axis 2	1.77	0.84	0.63	1.66	1.82
Ab10b	Axis 1	2.11	2.05	3.50	1.80	0.98
	Axis 2	3.40	3.39	3.58	2.62	3.48

Statistical Analysis					
Statistic	0"	1/2"	Center	1-1/2"	2"
Minimum Value	1.49	0.67	0.63	1.18	0.98
Maximum Value	3.40	3.39	3.58	2.62	3.48
Average Value	2.19	1.74	2.10	1.82	2.05
Standard Dev.	0.84	1.26	1.67	0.60	1.04
Variance	0.71	1.59	2.78	0.36	1.09
Conf. Limit, 90%	±0.99	±1.48	±1.96	±0.70	±1.22
Conf. Limit, 95%	±1.34	±2.01	±2.65	±0.95	±1.66
Sum of Squares	2.14	4.77	8.33	1.08	3.26

F-test Matrix					
s_1^2/s_2^2	0"	1/2"	Center	1-1/2"	2"
0"	n/a	2.23	3.90	1.99	1.53
1/2"	2.23	n/a	1.74	4.44	1.46
Center	3.90	1.74	n/a	7.74	2.55
1-1/2"	1.99	4.44	7.74	n/a	3.03
2"	1.53	1.46	2.55	3.03	n/a

Critical F (N=4, 95% confidence) = 9.28. All values are less than Critical F, so there is no statistically significant difference between the positions of the 4-day treated specimen. The data from all positions can be pooled.

There is much scatter in the absorption times at each position and from one specimen to another. However, the average absorption times are higher than those for the untreated specimen. The average absorption time for the 4-day treated specimen is higher than that of the untreated specimen.

Preliminary Microdrop Test: Cubes Immersed 4 Days

Chart: Absorption Time (minutes) vs. Position on Cross-Section (0", 1/2", Center, 1-1/2", 2")

Legend:
- Ab10a, Axis 1
- Ab10a, Axis 2
- Ab10b, Axis 1
- Ab10b, Axis 2
- Average Value
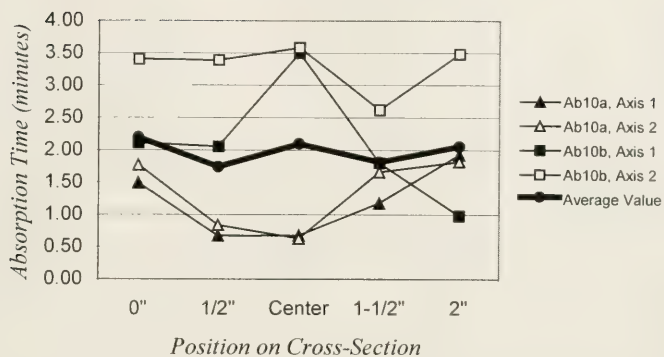
Pooled Data: Preliminary 4-day Treated Cubes	
Statistic	Value
Minimum Value	0.00
Maximum Value	2.63
Average Value	1.21
Standard Dev.	0.62
Variance	0.39
Conf. Limit, 90%	±0.24
Conf. Limit, 95%	±0.29
Sum of Squares	7.37
Within Treatment Mean Sq.	0.39

5-day Treated Specimen

Microdrop Absorption Time (minutes)						
Specimen		0"	1/2"	Center	1-1/2"	2"
Ab13a	Axis 1	3.76	2.23	3.56	1.59	3.50
	Axis 2	3.42	2.71	2.87	3.39	3.52
Ab13b	Axis 1	0.00	1.85	1.33	3.23	2.17
	Axis 2	3.86	2.30	1.99	1.60	1.58

Statistical Analysis					
Statistic	0"	1/2"	Center	1-1/2"	2"
Minimum Value	0.00	1.85	1.33	1.59	1.58
Maximum Value	3.86	2.71	3.56	3.39	3.52
Average Value	2.76	2.27	2.44	2.45	2.69
Standard Dev.	1.85	0.35	0.98	0.99	0.97
Variance	3.42	0.12	0.96	0.98	0.95
Conf. Limit, 90%	±2.17	±0.41	±1.15	±1.17	±1.14
Conf. Limit, 95%	±2.94	±0.56	±1.56	±1.58	±1.55
Sum of Squares	10.26	0.37	2.87	2.95	2.85

F-test Matrix					
s_1^2/s_2^2	0"	1/2"	Center	1-1/2"	2"
0"	n/a	27.55	3.57	3.47	3.60
1/2"	27.55	n/a	7.72	7.93	7.64
Center	3.57	7.72	n/a	1.03	1.01
1-1/2"	3.47	7.93	0.96	n/a	1.04
2"	3.60	7.64	1.01	1.04	n/a

Critical F (N=4, 95% confidence level) = 9.28. Those values highlighted are greater than Critical F. This means there is a statistically significant difference between 0" and 1/2" positions. The reason for this is unknown, since the other positions are not statistically different from each other. The data from all positions will be pooled.

There is much scatter in the absorption times at each position and from one specimen to another. The average absorption times for the 5-day treated specimen are quite higher than the untreated, 3-day treated, or 4-day treated specimen. This could indicate much more consolidant entered the specimen in 5 days of immersion.

Preliminary Microdrop Test:
5-Day Treated Cubes

Legend:
- Ab13a, Axis 1
- Ab13a, Axis 2
- Ab13b, Axis 1
- Ab13b, Axis 2
- Average Value

Y-axis: *Absorption Time (minutes)*
X-axis: *Position on Cross-Section* (0", 1/2", Center, 1-1/2", 2")
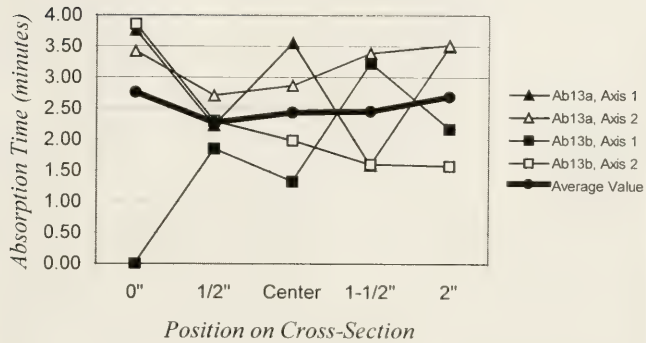
Pooled Data: Preliminary 5-day Treated Cubes	
Statistic	**Value**
Minimum Value	0.00
Maximum Value	3.86
Average Value	2.52
Standard Dev.	1.02
Variance	1.05
Conf. Limit, 90%	±0.40
Conf. Limit, 95%	±0.48
Sum of Squares	19.95
Within Treatment Mean Sq.	1.05

Analysis of Variance: Untreated and Treated Preliminary Specimen

Variation Within Treatments		
Specimen	**Average Time (Pooled Data)**	**Sum of Squares**
Untreated	1.21	7.37
Immersed 3 days	1.92	11.20
Immersed 4 days	1.98	20.17
Immersed 5 days	2.52	19.95

Overall Sum of Squares = 58.69

Within Treatment Mean Square = 0.77

Grand Average = 1.91

Between Treatment Sum of Squares = 17.34

Between Treatment Mean Square = 5.78

Ratio of Mean Squares = 7.48

Critical F = 2.92.

7.48> 2.92, which means there is a statistically significant difference between treatments. This indicates that the each of the treatments is different; that is, the treated specimen are in fact treated, and that 3-day treatment is different than 4-day treatment is different than 5-day treatment.

Final Microdrop Absorption Test
Batches B, D and F Cubes

Untreated Specimen

Microdrop Absorption Time (minutes)						
Specimen		0"	1/2"	Center	1-1/2"	2"
Db2a	Axis 1, Trial 1	0.75	0.57	0.70	1.58	0.93
	Axis 1, Trial 2	1.86	1.42	1.43	2.52	1.60
	Axis 1, Trial 3	2.70	2.13	2.07	3.98	3.75
	Axis 2, Trial 1	1.25	0.62	0.70	0.67	0.73
	Axis 2, Trial 2	3.10	2.22	1.55	1.15	0.88
	Axis 2, Trial 3	2.37	1.67	1.58	1.65	2.57
Db2b	Axis 1, Trial 1	0.77	0.72	0.97	0.48	1.48
	Axis 1, Trial 2	1.68	1.43	1.52	2.30	1.25
	Axis 1, Trial 3	2.90	2.12	2.28	2.03	3.22
	Axis 2, Trial 1	1.02	1.02	1.02	0.90	0.80
	Axis 2, Trial 2	1.73	1.23	2.82	0.90	0.57
	Axis 2, Trial 3	2.37	3.10	2.70	3.35	1.67
Fb15a	Axis 1, Trial 1	0.70	2.03	0.82	0.67	0.88
	Axis 1, Trial 2	0.77	2.57	2.42	0.90	1.38
	Axis 1, Trial 3	1.60	2.37	1.83	0.80	2.22
	Axis 2, Trial 1	0.62	0.77	0.75	1.17	0.75
	Axis 2, Trial 2	0.82	0.82	0.90	1.48	0.62
	Axis 2, Trial 3	3.30	0.77	1.50	2.77	2.65
Fb15b	Axis 1, Trial 1	0.33	0.83	0.98	1.07	0.78
	Axis 1, Trial 2	1.08	0.85	1.22	0.88	0.80
	Axis 1, Trial 3	1.93	1.50	1.53	2.20	1.80
	Axis 2, Trial 1	0.52	1.47	0.93	0.98	0.35
	Axis 2, Trial 2	1.18	1.85	1.37	1.52	1.75
	Axis 2, Trial 3	1.77	2.77	0.98	2.60	0.67

Statistical Analysis					
Statistic	**0"**	**1/2"**	**Center**	**1-1/2"**	**2"**
Minimum Value	0.33	0.57	0.70	0.48	0.35
Maximum Value	3.30	3.10	2.82	3.98	3.75
Average Value	1.55	1.54	1.44	1.61	1.42
Standard Dev.	0.87	0.74	0.63	0.92	0.90
Variance	0.76	0.55	0.40	0.86	0.81
Conf. Limit, 90%	0.30	0.26	0.22	0.32	0.31
Conf. Limit, 95%	0.37	0.31	0.27	0.39	0.38
Sum of Squares	17.55	12.74	9.14	19.67	18.52

F-Test Matrix					
s_1^2/s_2^2	**0"**	**1/2"**	**Center**	**1-1/2"**	**2"**
0"	n/a	1.38	1.92	1.12	1.06
1/2"	1.38	n/a	1.39	1.54	1.45
Center	1.92	1.39	n/a	2.15	2.03
1-1/2"	1.12	1.54	2.15	n/a	1.06
2"	1.06	1.45	2.03	1.06	n/a

Critical F (N=24, 95% confidence) = 2.12. The values highlighted are greater than Critical F, meaning there is a statistically significant difference between the center and 1-1/2" position on the untreated specimen. This difference could concrete heterogeneity or experimental error. Because these specimen are untreated, pooling data from all positions is justified.

There is some scatter in the readings at each position from specimen to specimen.

Averages over the 3 Trials for Untreated Cubes					
Specimen	0"	1/2"	Center	1-1/2"	2"
Db2a, Axis 1	1.77	1.37	1.40	2.69	2.09
Db2a, Axis 2	2.24	1.50	1.28	1.16	1.39
Db2b, Axis 1	1.78	1.42	1.59	1.60	1.98
Db2b, Axis 2	1.71	1.78	2.18	1.72	1.01
Fb15a, Axis 1	1.02	2.32	1.69	0.79	1.49
Fb15a, Axis 2	1.58	0.79	1.05	1.81	1.34
Fb15b, Axis 1	1.11	1.06	1.24	1.38	1.13
Fb15b, Axis 2	1.16	2.03	1.09	1.70	0.92
Average Value	1.55	1.54	1.44	1.61	1.42

Final Microdrop Test:
Untreated Cubes

189
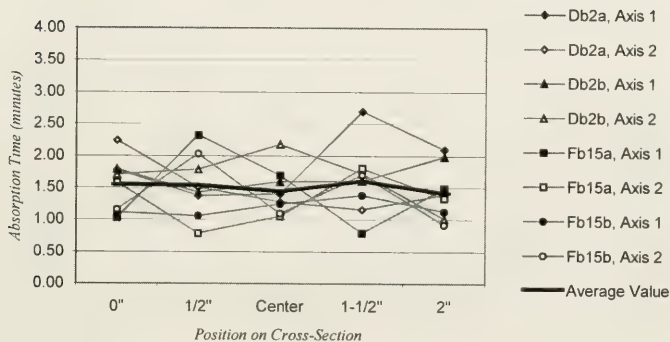
1-Hour Treated Cube Specimen

(Not all analysis is included because the results were used only for comparison.)

Averages over the 3 Trials for 1-Hour Treated Cube Specimen					
Specimen	**0"**	**1/2"**	**Center**	**1-1/2"**	**2"**
Fb11a, Axis 1	1.99	1.99	0.95	1.14	2.62
Fb11a, Axis 2	2.99	1.38	0.98	0.44	1.82
Fb11b, Axis 1	2.85	1.00	1.71	1.55	1.38
Fb11b, Axis 2	2.04	0.45	1.64	1.97	3.07
Fb12a, Axis 1	0.61	1.62	1.48	1.73	1.62
Fb12a, Axis 2	2.88	1.01	1.94	0.76	0.85
Fb12b, Axis 1	0.79	1.22	1.00	1.14	0.78
Fb12b, Axis 2	1.60	1.24	1.07	0.92	2.00
Average Value	1.97	1.24	1.35	1.21	1.77

**Final Microdrop Test:
1-Hour Treated Specimen**
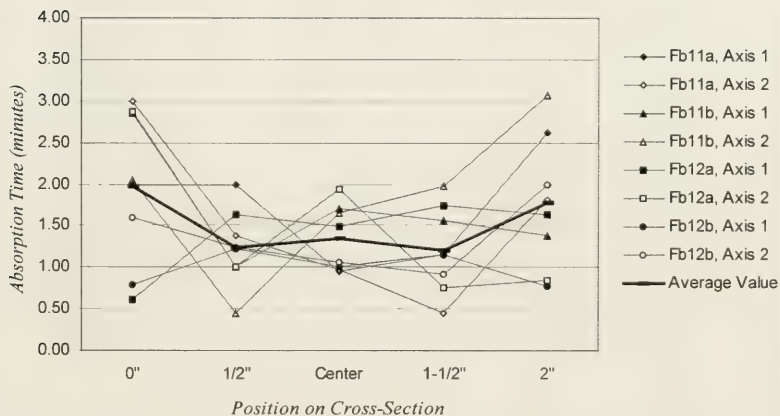
5-day Treated Specimen

	Microdrop Absorption Time (minutes)					
	Specimen	0"	1/2"	Center	1-1/2"	2"
Bb1a	Axis 1, Trial 1	1.23	1.20	0.32	0.40	0.67
	Axis 1, Trial 2	1.67	1.38	0.43	0.62	0.50
	Axis 1, Trial 3	2.73	1.83	1.82	0.25	3.57
	Axis 2, Trial 1	2.42	0.52	0.33	0.78	0.87
	Axis 2, Trial 2	4.72	0.73	1.12	1.17	3.10
	Axis 2, Trial 3	4.67	0.48	0.45	1.08	3.88
Bb1b	Axis 1, Trial 1	1.22	1.22	0.70	0.77	1.17
	Axis 1, Trial 2	3.10	2.37	2.00	1.45	1.20
	Axis 1, Trial 3	4.10	3.97	1.60	3.18	3.67
	Axis 2, Trial 1	1.97	0.93	0.70	0.75	1.37
	Axis 2, Trial 2	4.15	2.05	1.63	1.50	3.48
	Axis 2, Trial 3	5.05	2.47	1.23	2.53	4.53
Bb5a	Axis 1, Trial 1	1.97	3.08	0.65	1.93	1.35
	Axis 1, Trial 2	5.58	5.40	1.03	3.80	2.62
	Axis 1, Trial 3	0.83	4.22	0.98	2.23	4.27
	Axis 2, Trial 1	3.00	1.25	1.12	1.76	2.00
	Axis 2, Trial 2	3.53	1.03	0.83	3.37	3.70
	Axis 2, Trial 3	5.00	2.00	0.98	2.45	2.67
Bb5b	Axis 1, Trial 1	0.12	2.63	1.15	0.23	1.75
	Axis 1, Trial 2	0.12	2.33	2.58	1.23	3.12
	Axis 1, Trial 3	3.95	4.23	3.92	2.67	3.35
	Axis 2, Trial 1	2.52	1.58	1.68	1.67	0.75
	Axis 2, Trial 2	4.13	4.20	2.75	1.90	0.85
	Axis 2, Trial 3	3.53	1.90	2.57	3.15	2.60
Bb15a	Axis 1, Trial 1	2.55	2.05	0.90	0.93	2.18
	Axis 1, Trial 2	2.37	1.58	1.03	1.05	0.98
	Axis 1, Trial 3	3.50	2.07	1.57	2.42	2.18
	Axis 2, Trial 1	0.85	1.37	0.73	1.63	2.30
	Axis 2, Trial 2	3.50	2.55	1.57	1.97	1.12
	Axis 2, Trial 3	0.60	3.03	2.30	3.62	2.10
Bb15b	Axis 1, Trial 1	1.67	2.35	1.73	1.25	1.37
	Axis 1, Trial 2	3.88	2.26	2.45	1.32	4.75
	Axis 1, Trial 3	4.67	3.23	2.97	2.53	4.40
	Axis 2, Trial 1	1.60	1.57	0.83	1.35	1.72
	Axis 2, Trial 2	3.32	2.13	2.65	2.08	2.58
	Axis 2, Trial 3	3.75	3.58	1.95	2.48	4.62

Statistical Analysis					
Statistic	0"	1/2"	Center	1-1/2"	2"
Minimum Value	0.12	0.48	0.32	0.23	0.50
Maximum Value	5.58	5.40	3.92	3.80	4.75
Average Value	2.88	2.24	1.48	1.76	2.43
Standard Dev.	1.48	1.16	0.86	0.95	1.28
Variance	2.20	1.34	0.74	0.91	1.65
Conf. Limit, 90%	0.41	0.33	0.24	0.27	0.36
Conf. Limit, 95%	0.48	0.39	0.29	0.32	0.43
Sum of Squares	76.91	47.03	25.75	31.85	57.74

F-Test Matrix					
s_1^2/s_2^2	0"	1/2"	Center	1-1/2"	2"
0"	n/a	1.64	2.99	0.41	0.75
1/2"	1.64	n/a	1.83	0.68	1.23
Center	2.99	1.83	n/a	1.24	2.24
1-1/2"	0.41	0.68	1.24	n/a	0.55
2"	0.75	1.23	2.24	0.55	n/a

Critical F (N=36, 95% confidence) = 1.84. Those values highlighted are greater than Critical F, meaning there is a statistically significant difference between center and edge positions. This could indicate that the cross-section is not fully impregnated with consolidant.

Final Microdrop Test:
5-Day Treated Cubes

192
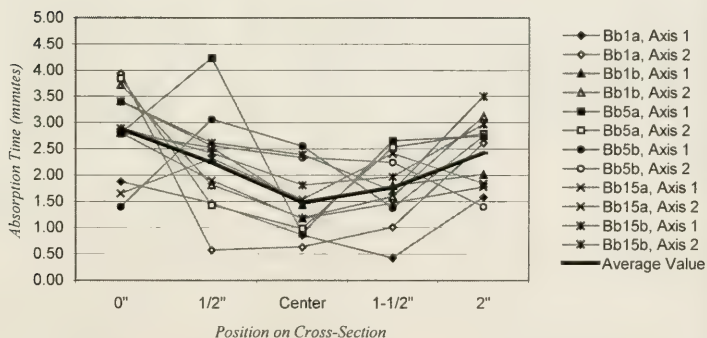
5-day Treated Specimen including 3/4" and 1-1/4" positions

	Specimen	0"	1/2"	3/4"	Center	1-1/4"	1-1/2"	2"
	Microdrop Absorption Time (minutes)							
Bb1a	Axis 1, Trial 1	1.23	1.20	1.52	0.32	1.55	0.40	0.67
	Axis 1, Trial 2	1.67	1.38	3.00	0.43	2.48	0.62	0.50
	Axis 1, Trial 3	2.73	1.83	1.32	1.82	1.33	0.25	3.57
	Axis 2, Trial 1	2.42	0.52	0.78	0.33	2.73	0.78	0.87
	Axis 2, Trial 2	4.72	0.73	2.35	1.12	3.62	1.17	3.10
	Axis 2, Trial 3	4.67	0.48	1.25	0.45	1.50	1.08	3.88
Bb1b	Axis 1, Trial 1	1.22	1.22	1.70	0.70	1.57	0.77	1.17
	Axis 1, Trial 2	3.10	2.37	2.83	2.00	2.87	1.45	1.20
	Axis 1, Trial 3	4.10	3.97	1.08	1.60	0.72	3.18	3.67
	Axis 2, Trial 1	1.97	0.93	4.67	0.70	2.75	0.75	1.37
	Axis 2, Trial 2	4.15	2.05	3.08	1.63	3.00	1.50	3.48
	Axis 2, Trial 3	5.05	2.47	1.17	1.23	1.08	2.53	4.53
Bb5a	Axis 1, Trial 1	1.97	3.08	2.18	0.65	3.72	1.93	1.35
	Axis 1, Trial 2	5.58	5.40	3.77	1.03	4.32	3.80	2.62
	Axis 1, Trial 3	0.83	4.22	1.38	0.98	2.50	2.23	4.27
	Axis 2, Trial 1	3.00	1.25	1.67	1.12	1.77	1.76	2.00
	Axis 2, Trial 2	3.53	1.03	2.88	0.83	3.90	3.37	3.70
	Axis 2, Trial 3	5.00	2.00	2.23	0.98	3.95	2.45	2.67
Bb5b	Axis 1, Trial 1	0.12	2.63	3.80	1.15	1.65	0.23	1.75
	Axis 1, Trial 2	0.12	2.33	4.83	2.58	4.92	1.23	3.12
	Axis 1, Trial 3	3.95	4.23	3.25	3.92	0.97	2.67	3.35
	Axis 2, Trial 1	2.52	1.58	4.38	1.68	4.63	1.67	0.75
	Axis 2, Trial 2	4.13	4.20	5.27	2.75	5.03	1.90	0.85
	Axis 2, Trial 3	3.53	1.90	0.78	2.57	1.28	3.15	2.60
Bb15a	Axis 1, Trial 1	2.55	2.05	1.32	0.90	0.67	0.93	2.18
	Axis 1, Trial 2	2.37	1.58	4.50	1.03	2.28	1.05	0.98
	Axis 1, Trial 3	3.50	2.07	0.72	1.57	1.03	2.42	2.18
	Axis 2, Trial 1	0.85	1.37	2.92	0.73	3.20	1.63	2.30
	Axis 2, Trial 2	3.50	2.55	3.97	1.57	3.87	1.97	1.12
	Axis 2, Trial 3	0.60	3.03	3.62	2.30	1.85	3.62	2.10
Bb15b	Axis 1, Trial 1	1.67	2.35	1.97	1.73	2.30	1.25	1.37
	Axis 1, Trial 2	3.88	2.26	3.82	2.45	3.75	1.32	4.75
	Axis 1, Trial 3	4.67	3.23	1.43	2.97	1.13	2.53	4.40
	Axis 2, Trial 1	1.60	1.57	4.53	0.83	2.50	1.35	1.72
	Axis 2, Trial 2	3.32	2.13	4.77	2.65	3.23	2.08	2.58
	Axis 2, Trial 3	3.75	3.58	3.20	1.95	1.77	2.48	4.62

Statistical Analysis							
Statistic	0"	1/2"	3/4"	Center	1-1/4"	1-1/2"	2"
Minimum Value	0.12	0.48	0.72	0.32	0.67	0.23	0.50
Maximum Value	5.58	5.40	5.27	3.92	5.03	3.80	4.75
Average Value	2.88	2.24	2.72	1.48	2.54	1.76	2.43
Standard Dev.	1.48	1.16	1.37	0.86	1.25	0.95	1.28
Variance	2.20	1.34	1.88	0.74	1.56	0.91	1.65
Conf. Limit, 90%	0.41	0.33	0.39	0.24	0.35	0.27	0.36
Conf. Limit, 95%	0.48	0.39	0.46	0.29	0.42	0.32	0.43
Sum of Squares	76.91	47.03	65.78	25.75	54.66	31.85	57.74

F-Test Matrix							
s_1^2/s_2^2	0"	1/2"	3/4"	Center	1-1/4"	1-1/2"	2"
0"	n/a	1.64	1.17	2.99	1.41	0.41	0.75
1/2"	1.64	n/a	1.40	1.83	1.16	0.68	1.23
3/4"	1.17	1.40	n/a	2.56	1.20	2.07	1.14
Center	2.99	1.83	2.56	n/a	2.12	1.24	2.24
1-1/4"	1.41	1.16	1.20	2.12	n/a	1.72	1.06
1-1/2"	0.41	0.68	2.07	1.24	1.72	n/a	0.55
2"	0.75	1.23	1.14	2.24	1.06	0.55	n/a

Critical F (N=36, 95% confidence) = 1.84. Those values highlighted are greater than Critical F, meaning there is a statistically significant difference between center and edge positions, and center, 3/4", and 1-1/4" positions. This could indicate that the cross-section is not fully impregnated with consolidant, but that the 3/4" and 1-1/4" positions are statistically "consolidated."

Analysis of Variance: Untreated and 5-day Treated Final Cube Specimen

Variance Within Treatments	
Specimen	**Sum of Squares**
Untreated	78.19
Immersed 5 days	239.28

Overall Sum of Squares = 317.47

Within Treatment Mean Square = 5.47

Grand Average = 1.9

Between Treatment Sum of Squares = 391.34

Between Treatment Mean Square = 391.34

Ratio of Mean Squares = 71.50

Critical F is less than 3.32 and 71.50>>3.32, meaning that there is a statistically significant difference between the untreated and 5-day treated specimen. The "treated" specimen are in fact treated.

Final Microdrop Absorption Test
Cylinder Cores

Untreated Cylinders

Microdrop Absorption Time (minutes)							
Specimen		0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
U1	Trial 1	2.15	3.43	1.77	2.87	5.00	3.26	2.38
U1	Trial 2	2.27	2.15	2.40	2.40	3.55	4.45	3.87
U1	Trial 3	1.25	1.98	3.52	3.20	4.23	4.75	3.65
U2	Trial 1	0.43	1.95	1.80	1.32	1.87	3.85	1.25
U2	Trial 2	1.22	1.17	2.53	0.65	3.58	1.67	5.3
U2	Trial 3	1.08	1.53	4.97	0.93	5.73	1.38	5.53
U3	Trial 1	0.37	1.33	1.78	1.80	3.12	0.93	1.5
U3	Trial 2	2.58	5.02	4.78	4.70	4.53	3.15	1.22
U3	Trial 3	1.83	5.00	4.88	4.92	4.68	3.5	3.55
U4	Trial 1	1.60	3.02	3.22	3.38	2.81	2.38	2.72
U4	Trial 2	2.57	2.27	5.77	3.75	4.90	2.43	3.85
U4	Trial 3	1.62	2.50	4.88	3.22	2.92	2.58	4.3

Statistical Analysis							
Statistic	0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
Minimum Value	0.37	1.17	1.77	0.65	1.87	0.93	1.22
Maximum Value	2.58	5.02	5.77	4.92	5.73	4.75	5.53
Average Value	1.58	2.61	3.53	2.76	3.91	2.86	3.26
Standard Dev.	0.75	1.29	1.47	1.39	1.12	1.19	1.47
Variance	0.56	1.67	2.17	1.92	1.25	1.41	2.16
Conf. Limit, 90%	±0.39	±0.67	±0.76	±0.72	±0.58	±0.62	±0.76
Conf. Limit, 95%	±0.47	±0.82	±0.93	±0.88	±0.71	±0.76	±0.93
Sum of Squares	6.12	18.41	23.82	21.14	13.77	15.56	23.72

F-Test Matrix							
s_1^2/s_2^2	0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
0"	n/a	3.01	3.89	3.45	2.25	2.54	3.88
1/4"	3.01	n/a	1.29	1.15	1.34	1.18	1.29
1/2"	3.89	1.29	n/a	1.13	1.73	1.53	1.00
3/4"	3.45	1.15	1.13	n/a	1.53	1.36	1.12
1"	2.25	1.34	1.73	1.53	n/a	1.13	1.72
1-1/4"	2.54	1.18	1.53	1.36	1.13	n/a	1.52
1-1/2"	3.88	1.29	1.00	1.12	1.72	1.52	n/a

Critical F (N=12, 95% confidence) = 2.92. Those values highlighted are greater than 2.92, meaning there is a statistically significance difference between some of the positions on the untreated core. This could represent concrete material heterogeneity or could show some damage of the cylinder during coring.

Treated Cylinders

Microdrop Absorption Time (minutes)								
Specimen		0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
T1	Trial 1	3.75	3.78	4.75	4.43	4.52	1.55	2.43
	Trial 2	5.92	3.57	6.50	4.42	3.98	1.68	3.55
	Trial 3	4.27	5.85	6.67	5.98	5.33	2.32	5.70
T2	Trial 1	4.78	3.23	5.42	2.95	3.75	5.03	1.52
	Trial 2	6.89	3.98	6.12	3.25	3.10	2.95	2.28
	Trial 3	5.02	6.00	5.87	4.60	3.67	4.25	1.65
T3	Trial 1	6.88	5.70	4.93	3.45	4.10	3.47	2.23
	Trial 2	7.25	4.23	5.87	3.08	4.50	2.38	3.73
	Trial 3	7.00	5.08	6.23	3.58	6.10	4.70	2.38
T4	Trial 1	7.83	5.05	4.33	2.75	3.70	1.87	1.95
	Trial 2	7.50	6.08	4.12	3.82	5.32	2.85	2.43
	Trial 3	7.50	6.50	3.53	3.92	4.35	2.98	3.73

Statistical Analysis							
Statistic	0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
Minimum Value	3.75	3.23	3.53	2.75	3.10	1.55	1.52
Maximum Value	7.83	6.50	6.67	5.98	6.10	5.03	5.70
Average Value	6.22	4.92	5.36	3.85	4.37	3.00	2.80
Standard Dev.	1.41	1.12	1.02	0.90	0.85	1.16	1.18
Variance	1.99	1.26	1.03	0.82	0.73	1.34	1.40
Conf. Limit, 90%	0.73	0.58	0.53	0.47	0.44	0.60	0.62
Conf. Limit, 95%	0.90	0.71	0.65	0.57	0.54	0.74	0.75
Sum of Squares	21.93	13.86	11.38	8.97	8.02	14.79	15.43

F-Test Matrix							
s_1^2/s_2^2	0"	1/4"	1/2"	3/4"	1"	1-1/4"	1-1/2"
0"	n/a	0.63	0.52	0.41	0.37	0.67	0.70
1/4"	0.63	n/a	0.82	0.65	1.73	0.94	1.11
1/2"	0.52	0.82	n/a	1.27	1.42	0.77	0.74
3/4"	0.41	0.65	1.27	n/a	1.12	0.61	1.72
1"	0.37	1.73	1.42	1.12	n/a	1.84	1.92
1-1/4"	0.67	0.94	0.77	0.61	1.84	n/a	1.04
1-1/2"	0.70	1.11	0.74	1.72	1.92	1.04	n/a

Critical F (n=12, 95% confidence) = 2.92. All values are less than Critical F, meaning there is no statistically significant difference between the positions.

Analysis of Variance: Untreated and Treated Cylinder Specimen

Variation Within Treatments			
Specimen	**Average Time**	**Sum of Squares**	**Mean Square**
Untreated	2.93	163.07	2.12
Treated, 0"	6.22	21.93	1.99
Treated, 1/4"	4.92	13.86	1.26
Treated, 1/2"	5.36	11.38	1.03
Treated, 3/4"	3.85	8.97	0.82
Treated, 1"	4.37	8.02	0.73
Treated, 1-1/4"	3.00	14.79	1.34
Treated, 1-1/2"	2.80	15.43	1.40

F-Test Matrix: Comparing Mean Squares								
	Untreated	**Treated 0"**	**Treated 1/4"**	**Treated 1/2"**	**Treated 3/4"**	**Treated 1"**	**Treated 1-1/4"**	**Treated 1-1/2"**
Untreated	n/a	1.06	1.68	2.05	2.60	2.91	1.58	1.51
Treated, 0"	1.06	n/a	1.58	1.93	2.44	2.73	1.48	1.42
Treated, 1/4"	1.68	1.58	n/a	1.22	1.55	1.73	1.07	1.11
Treated, 1/2"	2.05	1.93	1.22	n/a	1.27	1.42	1.30	1.36
Treated, 3/4"	2.60	2.44	1.55	1.27	n/a	1.12	1.65	1.72
Treated, 1"	2.91	2.73	1.73	1.42	1.12	n/a	1.84	1.92
Treated, 1-1/4"	1.58	1.48	1.07	1.30	1.65	1.84	n/a	1.04
Treated, 1-1/2"	1.51	1.42	1.11	1.36	1.72	1.92	1.04	n/a

When comparing any untreated to treated position: Critical F = 2.13. There is a statistically significant difference between the untreated and 3/4" and 1" positions.

When comparing any treated position to another treated position: Critical F= 2.92. There is no statistically significant difference between any of the treated positions.

Cylinders are treated to a depth of 1".

APPENDIX D: WEIGHT OF SPECIMEN

2" Concrete Cube Weight (g): Batch B								
	Before Treatment	**After Treatment**						
Sample/Days after Batching	**20 Jan 2000**	**25 Jan 2000**	**1 Feb 2000**	**4 Feb 2000**	**8 Feb 2000**	**14 Feb 2000**	**17 Feb 2000**	**22 Feb 2000**
	7	**12**	**19**	**22**	**26**	**32**	**35**	**40**
Bb1	312.32	314.21	312.40	0.00	0.00	0.00	0.00	0.00
Bb2	322.13	not treated	318.50	317.88	317.31	316.69	316.35	316.06
Bb3	322.55	not treated	319.24	318.56	317.95	317.33	316.96	316.67
Bb4	323.94	not treated	320.55	319.85	319.21	318.55	318.18	317.85
Bb5	325.21	327.46	325.71	0.00	0.00	0.00	0.00	0.00
Bb6	327.22	not treated	323.03	322.34	321.71	321.03	320.67	320.33
Bb7	320.35	321.37	319.35	318.70	318.03	317.19	316.77	316.32
Bb8	324.11	325.43	323.57	322.94	322.32	321.51	321.10	320.65
Bb9	319.53	not treated	315.72	315.05	314.46	313.79	313.44	313.14
Bb10	323.39	not treated	319.55	318.89	318.32	317.68	317.33	317.01
Bb11	323.25	not treated	319.60	318.97	318.36	317.74	317.42	317.13
Bb12	320.79	322.42	320.55	319.97	319.37	318.61	318.22	317.82
Bb13	321.38	not treated	317.38	316.70	316.11	315.48	315.16	314.87
Bb14	312.53	not treated	309.12	308.49	307.94	307.38	307.05	306.80
Bb15	307.82	310.13	308.45	0.00	0.00	0.00	0.00	0.00

2” Concrete Cube Weight (g): Batch D								
	Before Treatment	**After Treatment**						
Sample/Days after Batching	**21 Jan 2000**	**26 Jan 2000**	**1 Feb 2000**	**4 Feb 2000**	**8 Feb 2000**	**14 Feb 2000**	**17 Feb 2000**	**22 Feb 2000**
	7	**12**	**18**	**21**	**25**	**31**	**34**	**39**
Db1	320.35	321.38	320.02	319.51	318.95	318.29	317.94	317.58
Db2	310.53	not treated	307.30	0.00	0.00	0.00	0.00	0.00
Db3	318.21	not treated	314.74	314.09	313.53	312.90	312.57	312.27
Db4	320.04	321.09	319.62	319.06	318.52	317.80	317.43	317.06
Db5	322.13	not treated	318.97	318.34	317.80	317.19	316.86	316.57
Db6	321.17	not treated	317.98	317.32	316.77	316.13	315.83	315.55
Db7	318.18	318.99	317.54	316.97	316.41	315.69	315.35	314.95
Db8	317.76	318.49	316.97	316.40	315.83	315.13	314.76	314.40
Db9	320.40	321.47	320.03	319.48	318.95	318.25	317.89	317.54
Db10	319.28	not treated	316.36	315.76	315.23	314.64	314.32	314.04
Db11	315.07	316.77	315.31	314.78	314.26	313.57	313.22	312.88
Db12	320.23	not treated	316.86	316.22	315.68	315.06	314.73	314.49
Db13	321.02	322.52	320.89	320.33	319.74	319.00	318.65	318.27
Db14	314.21	not treated	310.87	310.26	309.73	309.12	308.82	308.58
Db15	322.40	not treated	319.04	318.39	317.80	317.17	316.86	316.58

2” Concrete Cube Weight (g): Batch F								
	Before Treatment	**After Treatment**						
Sample/Days after Batching	**22 Jan 2000**	**27 Jan 2000**	**1 Feb 2000**	**4 Feb 2000**	**8 Feb 2000**	**14 Feb 2000**	**17 Feb 2000**	**22 Feb 2000**
	7	**12**	**17**	**20**	**24**	**30**	**33**	**38**
Fb1	318.58	not treated	310.27	309.48	308.76	308.00	307.69	307.25
Fb2	322.53	not treated	319.12	318.31	317.60	316.85	316.45	316.11
Fb3	321.15	322.51	320.83	320.03	319.28	318.39	317.96	317.53
Fb4	321.08	not treated	317.51	316.74	316.06	315.32	314.94	314.61
Fb5	324.10	not treated	321.14	320.35	319.67	318.94	318.56	318.24
Fb6	315.79	not treated	312.20	311.38	310.69	309.92	309.54	309.22
Fb7	314.71	317.06	314.45	313.68	312.96	312.09	311.67	311.25
Fb8	319.92	not treated	315.51	314.70	314.02	313.29	312.91	312.59
Fb9	326.97	not treated	322.97	322.20	321.52	320.80	320.41	320.09
Fb10	320.15	321.85	320.23	319.50	318.82	318.01	317.62	317.23
Fb11	315.91	316.77	312.40	0.00	0.00	0.00	0.00	0.00
Fb12	319.57	320.44	316.13	0.00	0.00	0.00	0.00	0.00
Fb13	326.00	326.68	325.01	324.19	323.41	322.50	322.09	321.64
Fb14	324.11	325.63	323.98	323.17	322.40	321.52	321.11	320.70
Fb15	315.78	not treated	312.78	0.00	0.00	0.00	0.00	0.00

Consolidant Absorbed by Treated 2" Concrete Cubes Immersed for 5 Days				
Sample/Days after Batching	Weight Before Treatment	Weight After Treatment	Weight Gain (g)	Weight Gain (%)
Bb1	312.32	314.21	1.89	0.61
Bb5	325.21	327.46	2.25	0.69
Bb7	320.35	321.37	1.02	0.32
Bb8	324.11	325.43	1.32	0.41
Bb12	320.79	322.42	1.63	0.51
Bb15	307.82	310.13	2.31	0.75
Db1	320.35	321.38	1.03	0.32
Db4	320.04	321.09	1.05	0.33
Db7	318.18	318.99	0.81	0.25
Db8	317.76	318.49	0.73	0.23
Db9	320.40	321.47	1.07	0.33
Db11	315.07	316.77	1.70	0.54
Db13	321.02	322.52	1.50	0.47
Fb3	321.15	322.51	1.36	0.42
Fb7	314.71	317.06	2.35	0.75
Fb10	320.15	321.85	1.70	0.53
Fb11	315.91	316.77	0.86	0.27
Fb12	319.57	320.44	0.87	0.27
Fb13	326.00	326.68	0.68	0.21
Fb14	324.11	325.63	1.52	0.47

Statistical Analysis: Weight Gain (%)			
Statistic	Batch B	Batch D	Batch F
Minimum Value	0.32	0.23	0.21
Maximum Value	0.75	0.54	0.75
Average Value	0.55	0.35	0.42
Standard Dev.	0.17	0.11	0.19
Variance	0.03	0.01	0.03
Conf. Limit, 90%	0.14	0.08	0.14
Conf. Limit, 95%	0.18	0.10	0.17

APPENDIX E: COMPRESSION TESTING RESULTS

Preliminary Compression Testing

The load-displacement graph on the next page records the testing and shows the maximum load achieved by the specimen. The modulus of elasticity was determined by choosing two points on the linear portion of the load-displacement curve, finding the stress and strain of each point, and calculating the slope between them. The slope of the linear portion of the stress-strain curve is the modulus of elasticity.

Preliminary Compression Test: Untreated 2" Cubes			
Specimen	Maximum Load (lb)	Max. Comp. Stress (psi)	E (psi)
Ab2	17460	4365	166667
Ab3	26070	6517.5	250000
Ab4	24300	6075	214286
Average	**22610**	**5652.5**	**210317**

Preliminary Compression Test: 5-day Treated 2" Cubes			
Specimen	Maximum Load (lb)	Max. Comp. Stress (psi)	E (psi)
Ab7	19500	4875	187500
Ab8	24870	6217.5	250000
Ab9	23400	5850	214286
Average	**22590**	**5647.5**	**217262**
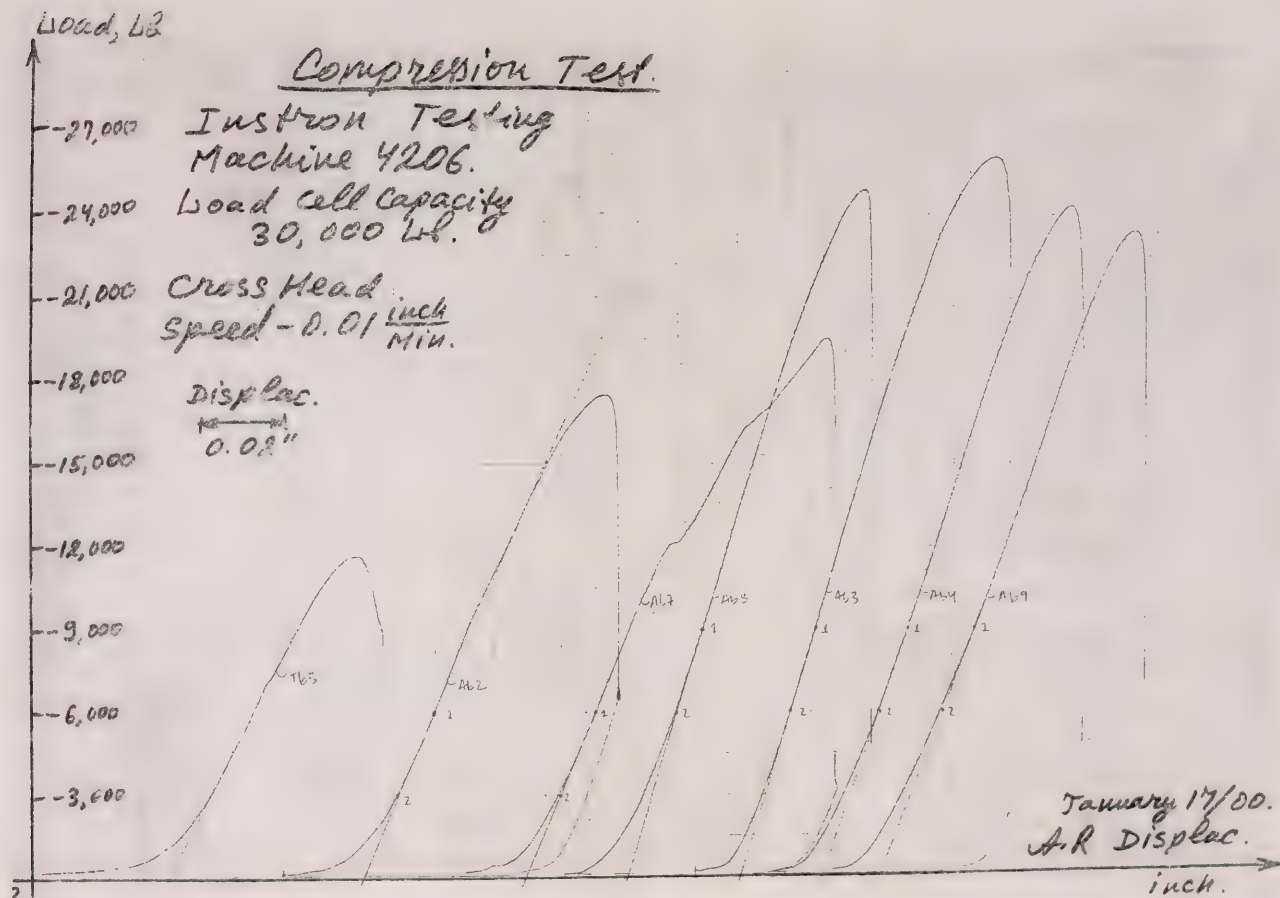
Final Compression Testing

Cubes

<u>Raw Data</u> "U" after specimen number indicates untreated cubes; "T" indicates treated cubes.

Bb4 U	Displacement (in)	Load (lb)
	0	0
	0.023	4000
	0.027	8000
	0.031	12000
	0.035	16000
	0.043	20000
	0.050	20400

Bb11 U	Displacement (in)	Load (lb)
	0	0
	0.016	4000
	0.019	8000
	0.021	12000
	0.023	16000
	0.026	20000
	0.031	23200

Bb6 U	Displacement (in)	Load (lb)
	0	0
	0.013	4000
	0.017	8000
	0.022	12000
	0.027	16000
	0.036	20000
	0.044	20900

Bb13 U	Displacement (in)	Load (lb)
	0	0
	0.008	4000
	0.010	8000
	0.013	12000
	0.015	16000
	0.019	20000
	0.025	22600

Db3 U	Displacement (in)	Load (lb)
	0	0
	0.014	4000
	0.018	8000
	0.020	12000
	0.021	16000
	0.023	20000
	0.026	24000
	0.032	26900

Db6 U	Displacement (in)	Load (lb)
	0	0
	0.006	4000
	0.010	8000
	0.012	12000
	0.017	16000
	0.022	20000
	0.027	24000
	0.033	25500

Graphing showed that Db6 did not behave linearly – the results are not included in the final analysis.

Db5 U	Displacement (in)	Load (lb)
	0	0
	0.048	4000
	0.053	8000
	0.056	12000
	0.059	16000
	0.063	20000
	0.069	24000
	0.073	24200

Fb1 U	Displacement (in)	Load (lb)
	0	0
	0.011	4000
	0.014	8000
	0.016	12000
	0.019	16000
	0.023	20000
	0.030	20300

Fb4 U	Displacement (in)	Load (lb)
	0	0
	0.019	4000
	0.022	8000
	0.025	12000
	0.028	16000
	0.032	20000
	0.045	22900

Fb2 U	Displacement (in)	Load (lb)
	0	0
	0.004	4000
	0.006	8000
	0.008	12000
	0.016	16000
	0.030	20000

Fb5 U	Displacement (in)	Load (lb)
	0	0
	0.009	4000
	0.012	8000
	0.017	12000
	0.021	16000
	0.035	19600

Fb6 U	Displacement (in)	Load (lb)
	0	0
	0.01	4000
	0.013	8000
	0.015	12000
	0.018	16000
	0.023	20000
	0.030	21000

Bb7 T	Displacement (in)	Load (lb)
	0	0
	0.042	4000
	0.047	8000
	0.050	12000
	0.054	16000
	0.062	19700

Bb8 T	Displacement (in)	Load (lb)
	0	0
	0	4000
	0.002	8000
	0.004	12000
	0.006	16000
	0.011	20000
	0.021	23100

Bb12 T	Displacement (in)	Load (lb)
	0	0
	0.005	4000
	0.007	8000
	0.008	12000
	0.009	16000
	0.011	20000
	0.019	27200

Db1 T	Displacement (in)	Load (lb)
	0	0
	0.032	4000
	0.038	8000
	0.041	12000
	0.045	16000
	0.050	20000
	0.059	23800

Db4 T	Displacement (in)	Load (lb)
	0	0
	0.035	4000
	0.037	8000
	0.039	12000
	0.040	16000
	0.042	20000
	0.043	24000
	0.047	26100

Db7 T	Displacement (in)	Load (lb)
	0	0
	0.013	4000
	0.015	8000
	0.017	12000
	0.019	16000
	0.020	20000
	0.022	24000
	0.027	28000
	0.031	29500

Db11 T	Displacement (in)	Load (lb)
	0	0
	0.027	4000
	0.029	8000
	0.031	12000
	0.032	16000
	0.033	20000
	0.035	24000
	0.037	28000
	0.045	30600

Db13 T	Displacement (in)	Load (lb)
	0	0
	0.021	4000
	0.023	8000
	0.025	12000
	0.026	16000
	0.028	20000
	0.031	24000
	0.037	26600

Fb3 T	Displacement (in)	Load (lb)
	0	0
	0.019	4000
	0.021	8000
	0.022	12000
	0.024	16000
	0.026	20000
	0.034	23900

Fb7 T	Displacement (in)	Load (lb)
	0	0
	0.010	4000
	0.013	8000
	0.016	12000
	0.020	16000
	0.024	20000
	0.030	21700

Fb10 T	Displacement (in)	Load (lb)
	0	0
	0.012	4000
	0.016	8000
	0.017	12000
	0.019	16000
	0.021	20000
	0.024	24000
	0.027	25400

Fb13 T	Displacement (in)	Load (lb)
	0	0
	0.032	4000
	0.038	8000
	0.040	12000
	0.041	16000
	0.042	20000
	0.044	24000
	0.057	25400

Analysis of Load

Untreated Cubes   **Treated Cubes**

Specimen	Max. Comp. Load (lb)
Bb4	20400
Bb6	20900
Bb11	23200
Bb13	22600
Average	21775
Std. Dev	1338
Variance	1789167
Conf. Limit, 95%	±2128
Sum of Squares	5367500

Specimen	Max. Comp. Load (lb)
Bb7	19700
Bb8	23100
Bb12	27200
Average	23333
Std. Dev	3755
Variance	14103333
Conf. Limit, 95%	±9330
Sum of Squares	28206667

Specimen	Max. Comp. Load (lb)
Db3	26900
Db5	24200
Db6	stress-strain not linear
	Db6 thrown out
Average	25550
Std. Dev	1909
Variance	3645000
Conf. Limit, 95%	±17153
Sum of Squares	3645000

Specimen	Max. Comp. Load (lb)
Db1	23800
Db4	26100
Db7	29500
Db11	30600
Db13	26600
Average	27320
Std. Dev	2734
Variance	7477000
Conf. Limit, 95%	±3395
Sum of Squares	29908000

Specimen	Max. Comp. Load (lb)
Fb1	20300
Fb2	20000
Fb4	22900
Fb5	19600
Fb6	21000
Average	20760
Std. Dev	1301
Variance	1693000
Conf. Limit, 95%	±1615
Sum of Squares	6772000

Specimen	Max. Comp. Load (lb)
Fb3	23900
Fb7	21700
Fb10	25400
Fb13	25400
Average	24100
Std. Dev	1749
Variance	3060000
Conf. Limit, 95%	±2783
Sum of Squares	9180000

F-Test Matrix: Untreated Cube Batches			
s_1^2/s_2^2	B	D	F
B	n/a	2.04	1.06
D	2.04	n/a	2.15
F	1.06	2.15	n/a

Critical F (95% Conf.), B-D >   9.55
Critical F (95% Conf.), B-F =   6.59
Critical F (95% Conf.), D-F >   6.94

F-Test Matrix: Treated Cube Batches			
s_1^2/s_2^2	B	D	F
B	n/a	1.89	4.61
D	1.89	n/a	2.44
F	4.61	2.44	n/a

Critical F (95% Conf.), B-D =   6.94
Critical F (95% Conf.), B-F =   9.55
Critical F (95% Conf.), D-F =   9.12

All values are less than Critical F. Can pool untreated batches and treated batches, respectively.

Statistics: Pooled Untreated Cubes	
Maximum Value	26900 lb
Minimum Value	19600 lb
Average Value	22000 lb
Standard Dev.	2211 lb
Variance	4888000
Conf. Limit, 90%	±1208
Conf. Limit, 95%	±1485
Sum of Squares	48880000

Statistics: Pooled Treated Cubes	
Maximum Value	30600 lb
Minimum Value	19700 lb
Average Value	25250 lb
Standard Dev.	3090 lb
Variance	9548182
Conf. Limit, 90%	±1677
Conf. Limit, 95%	±2058
Sum of Squares	105030000

209

Analysis of Variance: Untreated and Treated Cube Loading

Variation Within Treatments: Cubes	
Specimen	Sum of Squares
Untreated	48880000
Treated	105030000

Overall Sum of Squares =   153910000
Within Treatment Mean Sq. =   7329048

Grand average = 23696 lb

Between treatment sum of squares = 60619568

Between treatment mean square = 60619568

Ratio of mean Squares = 8.271

Critical F (95% conf.) = 3.864

8.271 > 3.864, so there is a statistically significant difference in the maximum load between treatments.

Analysis of Stress

Stress was calculated by dividing the maximum load by the cross-sectional area (4 in^2) of the cubes.

Untreated Cubes

Specimen	Max. Comp. Stress (psi)
Bb4	5100
Bb6	5225
Bb11	5800
Bb13	5650
Db3	6725
Db5	6050
Fb1	5075
Fb2	5000
Fb4	5725
Fb5	4900
Fb6	5250

Treated Cubes

Specimen	Max. Comp. Stress (psi)
Bb7	4925
Bb8	5775
Bb12	6800
Db1	5950
Db4	6525
Db7	7375
Db11	7650
Db13	6650
Fb3	5975
Fb7	5425
Fb10	6350
Fb13	6350

Statistics: Untreated Cubes		Statistics: Treated Cubes	
Maximum Value	6725 psi	Maximum Value	7650 psi
Minimum Value	4900 psi	Minimum Value	4925 psi
Average Value	5500 psi	Average Value	6313 psi
Standard Dev.	553	Standard Dev.	773
Conf. Limit, 90%	±302	Conf. Limit, 90%	±401
Conf. Limit, 95%	±371	Conf. Limit, 95%	±493

Because there was there was a statistically significant difference in the maximum load of the untreated and treated cubes, there must be a statistically significant difference in the stress of the untreated and treated cubes.

Analysis of Modulus of Elasticity

Untreated Cubes

Specimen	E (psi)
Bb4	500000
Bb6	428600
Bb11	1000000
Bb13	857100
Average	696425
Std. Dev	275855
Variance	76095749167
Conf. Limit, 95%	±438885
Sum of Squares	2.28287E+11

Treated Cubes

Specimen	E (psi)
Bb7	571400
Bb8	1000000
Bb12	2000000
Average	1190467
Std. Dev	733098
Variance	537432653333
Conf. Limit, 95%	±1821263
Sum of Squares	1.07487E+12

Specimen	E (psi)
Db3	1000000
Db5	625000
Average	812500
Std. Dev	265165
Variance	70312500000
Conf. Limit, 95%	±2382375
Sum of Squares	70312500000

Specimen	E (psi)
Db1	571400
Db4	1333300
Db7	1142900
Db11	1333300
Db13	1111100
Average	1098400
Std. Dev	312341
Variance	97556640000
Conf. Limit, 95%	±387760
Sum of Squares	3.90227E+11

Specimen	E (psi)
Fb1	750000
Fb2	1000000
Fb4	666700
Fb5	500000
Fb6	750000
Average	733340
Std. Dev	180659
Variance	±32637778000
Conf. Limit, 95%	224282
Sum of Squares	1.30551E+11

Specimen	E (psi)
Fb3	1176500
Fb7	571400
Fb10	1000000
Fb13	1333300
Average	1020300
Std. Dev	328781
Variance	±108096913333
Conf. Limit, 95%	523090
Sum of Squares	3.24291E+11

F-Test Matrix: Untreated Cube Batches			
s1^2/s2^2	B	D	F
B	n/a	1.08	2.33
D	1.08	n/a	2.15
F	2.33	2.15	n/a

Critical F (95% Conf.), B-D >   19.2
Critical F (95% Conf.), B-F =   6.59
Critical F (95% Conf.), D-F >   6.94

F-Test Matrix: Untreated Cube Batches			
s1^2/s2^2	B	D	F
B	n/a	5.51	4.97
D	5.51	n/a	1.11
F	4.97	1.11	n/a

Critical F (95% Conf.), B-D =   19.2
Critical F (95% Conf.), B-F =   9.55
Critical F (95% Conf.), D-F =   6.59

All values are less than Critical F. Can pool untreated batches and treated batches, respectively.

Untreated Cubes: Pooled Data   **Treated Cubes**

Statistics	
Maximum Value	1000000 psi
Minimum Value	428600 psi
Average Value	734309 psi
Standard Dev.	211453
Variance	44712400909
Conf. Limit, 90%	±115525
Conf. Limit, 95%	±142047
Sum of Squares	4.47124E+11

Statistics	
Maximum Value	2000000 psi
Minimum Value	571400 psi
Average Value	1095383 psi
Standard Dev.	408890
Variance	167190968788
Conf. Limit, 90%	±212465
Conf. Limit, 95%	±260742
Sum of Squares	1.8391E+12

Analysis of Variance: Untreated and Treated Cubes Modulus of Elasticity

Variance Within Treatments: Cubes	
Specimen	**Sum of Squares**
Untreated	4.47124E+11
Treated	1.8391E+12

Overall Sum of Squares =   2.28622E+12
Within Treatment Mean Sq. =   1.08868E+11

Grand average = 922696 psi

Between treatment sum of squares = 7.48E+11

Between treatment mean square = 7.48E+11

Ratio of mean squares = 6.87

Critical F (95% conf.) = 3.86

6.87 > 3.86, so there is a statistically significant difference in the modulii of elasticity between treatments.

Cylinders

Raw Data   "U" after specimen number indicates untreated cylinders; "T" indicates treated cylinders.

Bc1 U	Displacement (in)	Load (lb)
	0	0
	0.006	25000
	0.011	50000
	0.017	75000
	0.022	100000
	0.028	125000
	0.042	150000

Bc2 U	Displacement (in)	Load (lb)
	0	0
	0.017	25000
	0.023	50000
	0.027	75000
	0.032	100000
	0.038	125000
	0.046	136500

Bc5 U	Displacement (in)	Load (lb)
	0	0
	0.007	25000
	0.013	50000
	0.018	75000
	0.022	100000
	0.026	125000
	0.031	138500

Cc3 U	Displacement (in)	Load (lb)
	0.000	0
	0.008	25000
	0.014	50000
	0.019	75000
	0.024	100000
	0.029	125000
	0.036	150000
	0.040	154000

Cc4 U	Displacement (in)	Load (lb)
	0.000	0
	0.016	25000
	0.020	50000
	0.023	75000
	0.027	100000
	0.032	125000
	0.038	150000
	0.046	162000

Dc1 U	Displacement (in)	Load (lb)
	0.000	0
	0.008	25000
	0.014	50000
	0.018	75000
	0.022	100000
	0.027	125000
	0.033	145000

Ec1 U	Displacement (in)	Load (lb)
	0.000	0
	0.015	25000
	0.020	50000
	0.025	75000
	0.030	100000
	0.037	125000
	0.042	132000

Ec4 U	Displacement (in)	Load (lb)
	0.000	0
	0.031	25000
	0.036	50000
	0.041	75000
	0.046	100000
	0.053	125000
	0.066	142500

215

Ec5 U	Displacement (in)	Load (lb)
	0.000	0
	0.011	25000
	0.016	50000
	0.020	75000
	0.025	100000
	0.031	125000
	0.040	150000
	0.044	147500

Gc2 U	Displacement (in)	Load (lb)
	0.000	0
	0.007	25000
	0.012	50000
	0.017	75000
	0.021	100000
	0.027	125000
	0.036	137500

Fc2 U	Displacement (in)	Load (lb)
	0.000	0
	0.010	25000
	0.016	50000
	0.021	75000
	0.027	100000
	0.036	125000
	0.043	132500

Gc4 U	Displacement (in)	Load (lb)
	0.000	0
	0.008	25000
	0.013	50000
	0.018	75000
	0.025	100000
	0.033	125000
	0.045	133000

Fc5 U	Displacement (in)	Load (lb)
	0.000	0
	0.011	25000
	0.017	50000
	0.021	75000
	0.024	100000
	0.030	125000

Graphing showed that Fc5 did not behave linearly
– the results are not included in the final analysis.

Bc4 T	Displacement (in)	Load (lb)
	0	0
	0.011	25000
	0.016	50000
	0.021	75000
	0.025	100000
	0.031	125000
	0.039	150000
	0.046	158750

Dc4 T	Displacement (in)	Load (lb)
	0.000	0
	0.030	25000
	0.034	50000
	0.037	75000
	0.040	100000
	0.043	125000
	0.046	150000
	0.050	153000

Cc1 T	Displacement (in)	Load (lb)
	0	0
	0.012	25000
	0.016	50000
	0.020	75000
	0.023	100000
	0.028	125000
	0.034	150000
	0.042	158000

Dc5 T	Displacement (in)	Load (lb)
	0.000	0
	0.009	25000
	0.013	50000
	0.017	75000
	0.021	100000
	0.025	125000
	0.032	150000
	0.038	158750

Cc5 T	Displacement (in)	Load (lb)
	0.000	0
	0.015	25000
	0.020	50000
	0.025	75000
	0.030	100000
	0.037	125000
	0.045	150000
	0.060	163500

Ec2 T	Displacement (in)	Load (lb)
	0.000	0
	0.011	25000
	0.017	50000
	0.022	75000
	0.026	100000
	0.032	125000
	0.039	150000
	0.045	159500

Dc3 T	Displacement (in)	Load (lb)
	0.000	0
	0.007	25000
	0.014	50000
	0.019	75000
	0.024	100000
	0.029	125000
	0.038	150000
	0.046	156500

Ec3 T	Displacement (in)	Load (lb)
	0.000	0
	0.015	25000
	0.021	50000
	0.026	75000
	0.030	100000
	0.035	125000
	0.042	150000
	0.047	155500

217

	Displacement (in)	Load (lb)
	0.000	0
	0.012	25000
	0.020	50000
Fc1 T	0.027	75000
	0.031	100000
	0.037	125000
	0.040	131500

Graphing showed that Fc1 did not behave linearly – the results are not included in the final analysis.

Analysis of Load

Untreated Cylinders

Specimen	Max. Comp. Load (lb)
Bc1	150000
Bc2	136500
Bc5	138500
Average	141667
Std. Dev.	7286
Variance	53083333
Conf. Limit, 95%	±18100
Sum of Squares	106166667

Specimen	Max. Comp. Load (lb)
Cc3	154000
Cc4	162000
Average	158000
Std. Dev.	5657
Variance	32000000
Conf. Limit, 95%	±50824
Sum of Squares	32000000

Specimen	Max. Comp. Load (lb)
Dc1	145000
No statistics performed	

Treated Cylinders

Specimen	Max. Comp. Load (lb)
Bc4	157500
No statistics performed	

Specimen	Max. Comp. Load (lb)
Cc1	158000
Cc5	163500
Average	160750
Std. Dev.	3889
Variance	15125000
Conf. Limit, 95%	±34942
Sum of Squares	15125000

Specimen	Max. Comp. Load (lb)
Dc3	156500
Dc4	153000
Dc5	158750
Average	156083
Std. Dev.	2898
Variance	8395833
Conf. Limit, 95%	±7199
Sum of Squares	16791667

Specimen	Max. Comp. Load (lb)	Specimen	Max. Comp. Load (lb)
Ec1	132000	Ec2	159500
Ec4	142500	Ec3	155500
Ec5	147500		
Average	140667	Average	157500
Std. Dev.	7911	Std. Dev.	2828
Variance	62583333	Variance	8000000
Conf. Limit, 95%	±19654	Conf. Limit, 95%	±25412
Sum of Squares	125166667	Sum of Squares	8000000

Specimen	Max. Comp. Load (lb)	Specimen	Max. Comp. Load (lb)
Fc2	132500	Fc1	stress-strain not linear -
Fc5	stress-strain not linear		Fc1 thrown out
	Fc5 thrown out		
No statistics performed		No statistics performed	

Specimen	Ult. Comp. Load (lb)	Specimen	Ult. Comp. Load (lb)
Gc2	137500	Gc1	139500
Gc4	133000	Gc3	130500
		Gc5	132000
Average	135250	Average	134000
Std. Dev.	3182	Std. Dev.	4822
Variance	10125000	Variance	23250000
Conf. Limit, 95%	±28589	Conf. Limit, 95%	±11979
Sum of Squares	10125000	Sum of Squares	46500000

F-Test Matrix: Untreated Cylinder Batches				
s_1^2/s_2^2	B	C	E	G
B	n/a	1.66	1.18	5.24
C	1.66	n/a	1.96	3.16
E	1.18	1.96	n/a	6.18
G	5.24	3.16	6.18	n/a

Critical F (95% Conf.), B-C >   19
Critical F (95% Conf.), B-E =   19
Critical F (95% Conf.), B-G >   19
Critical F (95% Conf.), C-E >   19
Critical F (95% Conf.), C-G >   19
Critical F (95% Conf.), E-G >   19

F-Test Matrix: Treated Cylinder Batches				
s_1^2/s_2^2	C	D	E	G
C	n/a	1.80	1.89	1.54
D	1.80	n/a	1.05	2.77
E	1.89	1.05	n/a	2.91
G	1.54	2.77	2.91	n/a

Critical F (95% Conf.), C-D >   19
Critical F (95% Conf.), C-E>   19
Critical F (95% Conf.), C-G >   19
Critical F (95% Conf.), D-E >   19
Critical F (95% Conf.), D-G >   19
Critical F (95% Conf.), E-G >   19

All values are less than Critical F. Can pool untreated and treated batches, respectively.

Statistics: Pooled Untreated Cylinders	
Maximum Value	162000 lb
Minimum Value	132000 lb
Average Value	142583 lb
Standard Dev.	9426 lb
Variance	88856061
Conf. Limit, 90%	±4898
Conf. Limit, 95%	±6011
Sum of Squares	977416667

Statistics: Pooled Treated Cylinders	
Maximum Value	163500 lb
Minimum Value	130500 lb
Average Value	151295 lb
Standard Dev.	11605 lb
Variance	134685227
Conf. Limit, 90%	±6340
Conf. Limit, 95%	±7796
Sum of Squares	1346852273

Analysis of Variance: Untreated and Treated Specimen Loading

Variance Within Treatments: Cylinders	
Specimen	Sum of Squares
Untreated	977416667
Treated	1346852273

Overall Sum of Squares =   2324268939
Within Treatment Mean Sq. =   110679473

Grand Average = 146,750 lb

Between treatment sum of squares = 435593943

Between treatment mean square = 435593943

Ratio of mean squares = 3.94

Critical F (95% conf.) = 3.86

3.94 > 3.86, so there is a statistically significant difference in the maximum load of the treated and untreated cylinders.

Analysis of Stress

Stress was calculated by dividing the maximum load by the cross-sectional area (28.27 in^2) of the cylinders.

Untreated Cylinders

Specimen	Max. Comp. Stress (psi)
Bc1	5306
Bc2	4828
Bc5	4899
Cc3	5447
Cc4	5730
Dc1	5129
Ec1	4669
Ec4	5041
Ec5	5218
Fc2	4687
Gc2	4864
Gc4	4705

Treated Cylinders

Specimen	Max. Comp. Stress (psi)
Bc4	5571
Cc1	5589
Cc5	5784
Dc3	5536
Dc4	5412
Dc5	5615
Ec2	5642
Ec3	5501
Gc1	4935
Gc3	4616
Gc5	4669

Statistics: Untreated Cylinders	
Maximum Value	5730
Minimum Value	4669
Average Value	5044
Standard Dev.	333
Conf. Limit, 90%	±173
Conf. Limit, 95%	±213

Statistics: Treated Cylinders	
Maximum Value	5784
Minimum Value	4616
Average Value	5352
Standard Dev.	411
Conf. Limit, 90%	±224
Conf. Limit, 95%	±276

Because there was a statistically significant difference in the maximum load of the untreated and treated cylinders, there must be a statistically significant difference in the stress of the untreated and treated cylinders.

Analysis of Modulus of Elasticity

Untreated Cylinders   **Treated Cylinders**

Specimen	E (psi)
Bc1	1932800
Bc2	2021100
Bc5	2238600
Average	2064167
Std. Dev.	157383
Variance	24769463333
Conf. Limit, 95%	±390993
Sum of Squares	49538926667

Specimen	E (psi)
Bc4	2238600
No statistics performed	

Specimen	E (psi)
Cc3	2021100
Cc4	2883700
Average	2452400
Std. Dev.	609950
Variance	372039380000
Conf. Limit, 95%	±5480097.8
Sum of Squares	372039380000

Specimen	E (psi)
Cc1	2659400
Cc5	2122400
Average	2390900
Std. Dev.	379716
Variance	144184500000
Conf. Limit, 95%	±3411561
Sum of Squares	144184500000

Specimen	E (psi)
Dc1	2640300
No statistics performed	

Specimen	E (psi)
Dc3	2122400
Dc4	3537000
Dc5	2659400
Average	2772933
Std. Dev.	714101
Variance	509940653333
Conf. Limit, 95%	±1774069
Sum of Squares	1019881306667

Specimen	E (psi)	Specimen	E (psi)
Ec1	2122400	Ec2	1516000
Ec4	2122400	Ec3	2267500
Ec5	2267500		
Average	2170767	*Average*	1891750
Std. Dev.	83774	*Std. Dev.*	531391
Variance	7018003333	*Variance*	282376125000
Conf. Limit, 95%	±208122	*Conf. Limit, 95%*	±4774280
Sum of Squares	14036006667	*Sum of Squares*	282376125000

Specimen	E (psi)	Specimen	E (psi)
Fc2	1868300	Fc1	
Fc5			
No statistics performed		*No statistics performed*	

Specimen	E (psi)	Specimen	E (psi)
Gc2	2267500	Gc1	2456500
Gc4	2131300	Gc3	2021100
		Gc5	2267500
Average	2199400	*Average*	2248367
Std. Dev.	96308	*Std. Dev.*	218330
Variance	9275220000	*Variance*	47667853333
Conf. Limit, 95%	±865279	*Conf. Limit, 95%*	±542405
Sum of Squares	9275220000	*Sum of Squares*	95335706667

Statistics: Pooled Untreated Cylinders		Statistics: Pooled Treated Cylinders	
Maximum Value	2883700 psi	Maximum Value	3537000 psi
Minimum Value	1868300 psi	Minimum Value	1516000 psi
Average Value	2209750 psi	Average Value	2351618 psi
Standard Dev.	291230 psi	Standard Dev.	504489 psi
Variance	84815077273	Variance	254509635636
Conf. Limit, 90%	±151328	Conf. Limit, 90%	±275622
Conf. Limit, 95%	±185713	Conf. Limit, 95%	±338900
Sum of Squares	932965850000	Sum of Squares	2545096356364

225

Analysis of Variance: Untreated and Treated Cylinders Modulus of Elasticity

Variance Within Treatments: Cylinders	
Specimen	Sum of Squares
Untreated	932965850000
Treated	2545096356364

Overall Sum of Squares =   3478062206364

Within Treatment Mean Sq. =   1.65622E+11

Grand Average = 2,267,500 psi

Between treatment sum of squares = 1.18E+11

Between treatment mean square = 1.18E+11

Ratio of mean squares = 0.71

Critical F = 3.86

0.71 < 3.86, so there is not a statistically significant difference in the modulii of elasticity of the untreated and treated cylinders.

BIBLIOGRAPHY

General References

Ashurst, John & Nicola. *Practical Building Conservation, English Heritage Technical Handbook Volume 1: Stone Masonry.* New York: Halstead Press, 1988.

Binda, Linda, and A. Anzani. "Structural Behavior and Durability of Stone Masonry." In *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage, 113-150. West Sussex, England: John Wiley & Sons Ltd, 1997.

D'Avino, Stefano. "Chemicals and Physical Techniques for the Conservation of Stone Materials: Interdisciplinary Approach and Specific Nature of the Discipline." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October* 1996, edited by Josef Riederer, Volume 2: 923-927. Berlin: moller druck und verlag gmbh, 1996.

ICOMOS. "The Venice Charter (1964)." Internet: www.international.icomos.org/icomos/e_venice.htm.

_____. "The Athens Charter for the Restoration of Historic Monuments (1931)." Internet: ww.icomos.org/docs/athens_charter.html, 1931.

_____. "The Australia ICOMOS Charter for the Conservation of Places of Cultural Significance (The Burra Charter, 1996)." Internet: www.icomos.org/docs/burra_charter.html.

ICOMOS International Committee on Analysis and Restoration of Structures of Architectural Heritage. "Recommendations for the Analysis, Conservation and Structural Restoration of Architectural Heritage." *US/ICOMOS Newsletter* (November-December 1999).

Ireson, A.S. *Masonry Conservation & Restoration.* Bristol, UK: J.W. Arrowsmith Ltd, 1987.

Rizzi, Gionata. "Stone Preservation and Architectural Conservation: Two Different Perspectives?" In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October* 1996, edited by Josef Riederer, Volume 2: 879-884. Berlin: moller druck und verlag gmbh, 1996.

Sabnis, Gajanan Sabnis, ed. *Rehabilitation, Renovation and Preservation of Concrete and Masonry Structures.* Detroit, MI: American Concrete Institute, 1985.

Searls, C.L. "Group Report: How Can We Diagnose the Condition of Stone Monuments and Arrive at Suitable Treatment Programs?" In *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage, 199-221. West Sussex, England: John Wiley & Sons Ltd, 1997.

Teutonico, J.M, et al. "Group Report: How Can We Ensure the Responsible and Effective Use of Treatments (Cleaning, Consolidation, Protection)?" In *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage, 293-313. West Sussex, England: John Wiley & Sons Ltd, 1997.

Torraca, Giorgio. "The Scientist's Role in Historic Preservation with Particular Reference to Stone Conservation." In *Conservation of Historic Stone Buildings and Monuments,* 13-21. Washington, DC: National Academy Press, 1982.

Wenzel, F. "The Civil Engineer in Monument Preservation: Continuity and Change of Tasks." In *Structural Conservation of Masonry, International Technical Conference, Athens, 3 October - 2 November 1989,* 391-397. Rome: ICCROM, 1990.

Zador, Dr. Mihaly. "Activity of the RILEM 25 PEM - ICOMOS Working Group: Its Utilization and Instructions in Monuments Restoration." In *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978.

Stone Geology and Deterioration

D'Ossat, Guglielmo De Angelis. *Guide to the Methodical Study of Monuments and Causes of Their Decay.* Rome: ICCROM, 1982.

Gauri, K., D.J. Hagerty, and C.R. Ullrich. "Comparative Physical Properties of Weathered Impregnated and Unimpregnated Marble." *Engineering Geology* 6 (1972): 235-250.

Grimmer, Anne E. *A Glossary of Historic Masonry Deterioration Problems and Preservation Treatments.* Washington, DC: Department of the Interior, National Parks Service, 1984.

Kehew, Alan E. *Geology for Engineers & Environmental Scientists,* 2nd ed. Englewood Cliffs, NJ: Prentice Hall, 1995.

Keller, W.D. "Progress and Problems in Rock Weathering Related to Stone Decay." In *Decay and Preservation of Stone, Engineering Geology Case Histories Number 11,* edited by Erhard M. Winkler, 37-46. Boulder, CO: The Geological Society of America, 1978.

Lewin, S.Z., and A.E. Charola. "Stone Decay Due to Foreign Inclusions." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 205-217. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Mamillan, M. "Methods for Assessing the Deterioration of Stone Monuments." In *Science, Technology and European Cultural Heritage, Proceedings of the European Symposium, Bologna, Italy, 13-16 June 1989,* edited by N.S. Baer, C. Sabbioni, and A.I. Sors, 90-99. London: Butterworth-Heinemann Ltd., 1991.

Medgyesi, I., M. Bleuer, and G. Kraisell. "Field Examination of Load Bearing Stone Structures." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 503-511. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Oettingen, Dieter Von. "Decay and Conservation of Natural Stone." *Building Stone Magazine,* September-October 1989, 6-8.

Rodrigues, J. Delgado. "Causes, Mechanisms and Measurement of Damage in Stone Monuments." In *Science, Technology and European Cultural Heritage, Proceedings of the European Symposium,*

Bologna, Italy, 13-16 June 1989, edited by N.S. Baer, C. Sabbioni, and A.I. Sors, 124-137. London: Butterworth-Heinemann Ltd., 1991.

Stambolov, T., and J.R.J. van Asperen de Boer. *The Deterioration and Conservation of Porous Building Materials in Monuments: A Literature Review,* 2nd ed. Rome: International Centre for the Study of Preservation and Restoration of Cultural Property, 1976.

Turkington, A.V. "Stone Durability." In *Processes of Urban Decay, Proceedings of SWAPNET '95,* edited by Bernard J. Smith and Patricia A. Warke, 19-31. London: Donhead Publishing, Ltd., 1996.

Warke, P.A. "Inheritance Effects in Building Stone Decay." In *Processes of Urban Decay, Proceedings of SWAPNET '95,* edited by Bernard J. Smith and Patricia A. Warke, 32-43. London: Donhead Publishing, Ltd., 1996.

Winkler, E.M. *Stone: Properties, Durability in Man's Environment.* New York: Springer-Verlag, 1975.

Winker, Erhard M. "Problems in the Deterioration of Stone." In *Conservation of Historic Stone Buildings and Monuments,* 108-119. Washington, DC: National Academy Press, 1982.

Zezza, U. "Influence of Mechanical Anisotropic Behaviour to Tensile Strength on Decay Evolution of Marbles in Monuments." In *Conservation of Stone and Other Materials, Proceedings of the International RILEM/UNESCO Congress, Paris, 29 June-1 July 1993,* edited by M.-J. Thiel, 220-227. London: E & FN Spon, 1993.

Masonry Treatments and Repair

Accardo, G., R. Cassano, P. Rossi-Doria, P. Sammuri, and M. Tabasso. "Screening of Products and Methods for the Consolidation of Marble." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 721-733. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Accardo, G., S. Massa, P. Rossi-Doria, P. Sammuri, and M. Tabassa. "Artificial Weathering of Carrara Marble: Relationships Between the Induced Variations of Some Physical Properties." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 243-273. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Alessi, Paolo, A. Cortesi, G. Torriano, and D. Visintin. "Problems Connected with Protection of Monuments from Aggressive Agents." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 1st International Symposium, Bari, 7-10 June 1989,* edited by F. Zezza, 383-387. Brescia, Italy: Grafo, 1990.

American Society for Testing and Materials. "Guidelines for the Selection and Use of Stone Consolidants, Draft No. 11." Philadelphia, PA: ASTM, September 1998 (draft).

Amoroso, G.G., and V. Fassina. *Stone Decay and Conservation: Atmospheric Pollution, Cleaning, Consolidation and Protection (Materials Science Monographs, 11).* New York: Elsevier Science Publishers B.V., 1983.

Applonia, L., and the NORMAL Commission "Protectives Experimentation Subgroup." "Methodology for the Evaluation of Protective Products for Stone Materials. Part II: Experimental Tests on Treated Samples." In *ICCROM International Colloquium: Methods of Evaluation Products for the*

Conservation of Porous Building Materials in Monuments, 19-21 June 1995. Rome: ICCROM, 1995.

Ashton, H.E. "Coatings for Masonry Surfaces." In *The Association for Preservation Technology Masonry Conservation and Cleaning Handbook,* edited by Keith Blades, Gail Sussman, and Martin Weaver, 229-232. (location not given): The Association for Preservation Technology, 1984.

Ashurst, John. "Cleaning and Surface Repair." In *Conservation of Historic Stone Buildings and Monuments,* 272-280. Washington, DC: National Academy Press, 1982.

Biscontin, Guido, and Gianna Riva. "Study about Water Vapour Permeability in Stone Materials after Protective Treatment." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 689-697. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Boos, M., J. Grobe, G. Hilbert, and J. Mler-Rochholz. "Modified Elastic Silicic-Acid Ester Applied on Natural Stone and Tests of Their Efficiency." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 3: 1179-1185. Berlin: moller druck und verlag gmbh, 1996.

Brackin, Anne E. "A Comparative Study of the Effects of Applying Acrylics and Silanes in Sequence and in Mixture, with a Case Study of The Column in the Convento of Mission San Jose Y San Miguel De Aguayo, Texas." *A Thesis in Historic Preservation Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of the Requirements for the Degree of Master of Science.* 1994.

Bradley, S.M. "Evaluation of Organo Silanes for Use in Consolidation of Sculpture Displayed Indoors." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 759-767. Lausanne, Switzerland: Presse polytechniques romandes, 1985.

Brus, Jiri, and Petr Kotlik. "Consolidation of Stone by Mixtures of Alkoxysilane and Acrylic Polymer." *Studies in Conservation* 41 (1996): 109-119.

_____. "Cracking of Organosilicone Stone Consolidants in Gel Form." *Studies in Conservation* 41 (1996): 55-59.

Caselli, A., and D. Kagi. "Methods Used to Evaluate the Efficacy of Consolidants on an Australian Sandstone." In *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995,* 121-130. Rome: ICCROM, 1995.

Charola, A.E. "Brief Introduction to Silanes, Siloxanes, Silicones and Silicate Esters." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 313-314. Paris: UNESCO, 1988.

Charola, A. Elena. "Laboratory Tests and Evaluation of Proposed Masonry Treatments." *APT Bulletin* 26 (1995): 35-39.

_____. "Water-Repellent Treatments for Building Stones: A Practical Overview." *APT Bulletin* 26 (1995): 10-17.

Charola, A.E., G.E. Wheeler, and G.G. Freund. "The Influence of Relative Humidity in the Polymerization of Methyl Trimethoxy Silane." In *Preprints of the IIC Congress "Adhesives and Consolidants,"* 177-181. Paris, 1984.

Charola, A.E., G.E. Wheeler, and R.J. Koestler. "Treatment of the Abydos Reliefs: Preliminary Investigations." In *4th International Congress on the Deterioration and Preservation of Stone Objects, Louisville, Kentucky, 7-9 July 1982,* edited by K.L. Gauri and J.A. Gwinn, 77-88. Louisville, KY: The University of Louisville, 1982.

Charola, A.E., R. Rossi-Manaresi, R.J. Koestler, G.E. Wheeler, and A. Tucci. "SEM Examination of Limestones Treated with Silanes or Prepolymerized Silicone Resin in Solution." In *Preprints of the IIC Congress "Adhesives and Consolidants,"* 182-184. Paris, 1984.

Christaras, Basile. "Particularities in Studying the Physical and Mechanical Properties of Stones in Monuments. Examples from the Mediterranean Basin." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October* 1996, edited by Josef Riederer, Volume 2: 819-829. Berlin: moller druck und verlag gmbh, 1996.

Clifton, James R., and Geoffrey J.C. Frohnsdorff. "Stone-Consolidating Materials: A Status Report." , In *Conservation of Historic Stone Buildings and Monuments,* 287-311. Washington, DC: National Academy Press, 1982.

Clifton, James R., and McClure Godette. "Performance Tests for Stone Consolidants." In *4th International Congress on the Deterioration and Preservation of Stone Objects, Louisville, Kentucky, 7-9 July 1982,* edited by K.L. Gauri and J.A. Gwinn, 101-108. Louisville, KY: The University of Louisville, 1982.

Crafts Council. *Science for Conservators Book 3: Adhesives and Coatings.* London: Museums & Galleries Commission, 1987.

De Witte, E., A.E. Charola, and R.P. Sherryl. "Preliminary Tests on Commercial Stone Consolidants." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 709-718. Lausanne, Switzerland: Presses Polytechniques Romandes, 1985.

Esbert, Rosa Maria, C.M. Grossi, L. Valdeon, Jorge Ordaz, F.J. Alonso, and R.M. Marcos. "Studies for Stone Conservation at the Cathedral of Murcia (Spain)." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 1st International Symposium, Bari, 7-10 June 1989,* edited by F. Zezza, 437-441. Brescia, Italy: Grafo, 1990.

Goins, E.S., G.S. Wheeler, and M.T. Wypyski. "Alkoxysilane Film Formation on Quartz and Calcite Crystal Surfaces." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 3: 1255-1264. Berlin: moller druck und verlag gmbh, 1996.

Goins, Elizabeth Stevenson, G.S. Wheeler, and S.A. Fleming. "The Influence of Reaction Parameters on the Effectiveness of Tetraethoxysilane-Based Stone Consolidants: Solvent Effects." In *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995,* 259-274. Rome: ICCROM, 1995.

Hanna, S.B. "The Use of Organo-Silanes for the Treatment of Limestone in an Advanced State of Deterioration." In *Adhesives and Consolidants, Preprints of the Contributions to the Paris*

Congress, 2-8 September 1984, 171-176. London: International Institute for Conservation of Historic and Artistic Works, 1984.

Honsinger, D., and H.R. Sasse. "Alteration of Microstructure and Moisture Characteristics of Stone Materials Due to Impregnation." In *Durability of Building Materials and Components, Proceedings of the Fifth International Conference held in Brighton, UK, 7-9 November 1990,* edited by J.M. Baker, P.J. Nixon, A.J. Majumdar and H. Davies, 213-224. London: E. & F.N. Spon, 1991.

Horie, C.V. *Materials for Conservation.* London: Butterworths, 1987.

Hristova, Julia, and V. Todorov. "Consolidation Effect of Wacker-Silicones on the Properties of Sandy Limestone." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 3: 1195-1201. Berlin: moller druck und verlag gmbh, 1996.

Kimmel, Jocelyn. "Characterization and Consolidation of Pennsylvania Blue Marble, with a Case Study of the Second Bank of the United States, Philadelphia, PA." *A Thesis in Historic Preservation Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of the Requirements for the Degree of Master of Science.* 1996.

Klemm, Dietrich D., and Rolf Snethlage. "Research on Surface-Conservation of Corroded Pentelish Marble of the Akropolis in Athens." In *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978.

Koblischek, Peter J. "Polymers in the Renovation of Buildings Constructed of Natural Stone in the Mediterranean Basin." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 3rd International Symposium, Venice, 22-25 June 1994,* edited by V. Fassina, H. Ott and F. Zezza, 849-854. Venice: Soprintendenza ai Beni Artistici e Storici di Venezia, 1994.

_____. "The Consolidation of Natural Stone with a Stone Strengthenr on the Basis of Poly-Silicic-Acid-Ethylester." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 3: 1187-1193. Berlin: moller druck und verlag gmbh, 1996.

Lewin, S.Z. "Recent Experience with Chemical Techniques of Stone Preservation." In *The Treatment of Stone,* 139-144. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1972.

_____. "The Current State of the Art in the Use of Synthetic Materials For Stone Conservation: Inorganic and Metal-Organic Compounds." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 290-302. Paris: UNESCO, 1988.

Lewin, S.Z., and A.D. Papdimitriou. "Investigation of Polymer Impregnation of Stone II: Solvent Transport of Pre-Polymerized Methyl Methacrylate." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 605-623. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Lewin, S.Z., and G.E. Wheeler. "Alkoxysilane Chemistry and Stone Conservation." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 831-844. Lausanne, Switzerland: Presse polytechniques romandes, 1985.

Littman, K., H.R. Sasse, S. Wagener, and H. Hocker. "Development of Polymers for the Consolidation of Natural Stone." In *Conservation of Stone and Other Materials, Proceedings of the International RILEM/UNESCO Congress, Paris, 29 June-1 July 1993,* edited by M.-J. Thiel, 681-688. London: E & FN Spon, 1993.

Lotzmann, Susanne, and H.R. Sasse. "Drilling Resistance as an Indicator for Effectiveness of Stone Consolidation." In *The Use of and Need for Preservation Standards in Architectural Conservation, ASTM STP 1355,* edited by L.B. Sickels-Taves, 77-89. West Conshohocken, PA: ASTM, 1999.

Lukaszewics, J.W. "Application of Silicone Products in the Conservation of Volcanic Tuffs." In *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990,* edited by A. Elena Charola, 191-202. Rome: ICCROM, 1994.

_____. "The Influence of Stone Pre-Consolidation with Ethyl Silicate on Deep Consolidation." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 3: 1209-1213. Berlin: moller druck und verlag gmbh, 1996.

Lukaszewicz, J.W., D. Kwiatkowski, and M. Klingspor. "Consolidation of Gotland Stone in Monuments." In *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995,* 179-187. Rome: ICCROM, 1995.

Mack, Robert C. "The Cleaning and Waterproof Coating of Masonry Buildings." *Preservation Brief No. 1.* Internet: www2.cr.nps.gov/tps/briefs, (1977).

Marchesini, L., and R. Bonora. "The Use of Silicon Resin in the Conservation and Protection of Stone." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 317-328. Paris: UNESCO, 1988.

Marinelli, Giuseppe. "Use of an Epoxy Aliphatic Resin in the Consolidation of Porous Building Materials Having Poor Mechanical Properties." In *The Conservation of Stone I, Proceedings of the International Symposium, Bologna, 19-21 June 1975,* 573-591. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1976.

Mavrov, George. "Aging of Silicone Resins." *Studies in Conservation* 28 (1983): 171-178.

Moncrieff, Anne. "The Treatment of Deteroriating Stone with Silicone Resins: Interim Report." *Studies in Conservation* 21 (1976): 179-191.

Munsch, Guy. "The First Bank of the United States, Reevaluating Success: A Conservation Conditions Assessment Survey and Analysis of Previous Treatments." *A Thesis in Historic Preservation Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of the Requirements for the Degree of Master of Science.* 1998.

Navarro, A., Joan Parrot, Montserrat Argemi, Jose Luis Prada, and Aureli Alvarez. "The Interface Behavior of the Natural Stone 'Lorito' (Tarragona) and the Synthetic Mortar 'Parrot's Mix 4' (I.Q. Parrot, S.A)." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the*

3rd International Symposium, Venice, 22-25 June 1994, edited by V. Fassina, H. Ott and F. Zezza, 625-629. Venice: Soprintendenza ai Beni Artistici e Storici di Venezia, 1994.

Ozturk, Isil. "Alkoxysilanes Consolidation of Stone and Earthen Building Materials." *A Thesis in Historic Preservation Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of the Requirements for the Degree of Master of Science.* 1992.

Pascua, N.G., M.I Sanchez de Rojas, and M. Frias. "Determination of the Differences in the Physical Properties of the Treated Villamayor Sandstone under Natural and Artificial Ageing." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 3rd International Symposium, Venice, 22-25 June 1994,* edited by V. Fassina, H. Ott and F. Zezza, 937-945. Venice: Soprintendenza ai Beni Artistici e Storici di Venezia, 1994.

Pascua, N. Garcia, M.I. Sanchez de Rojas, and M. Frias. "Study of Porosity and Physical Properties as Methods to Establish the Effectiveness of Treatments Used in Two Different Spanish Stones: Limestone and Sandstone." In *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995,* 147-162. Rome: ICCROM, 1995.

Price, C.A. "Brethane Stone Preservative." In *The Association for Preservation Technology Masonry Conservation and Cleaning Handbook,* edited by Keith Blades, Gail Sussman, and Martin Weaver. location not given: The Association for Preservation Technology, 1984.

_____. "Report on Session VI (Weathering)." In *ICCROM International Colloquium: Methods of Evaluating Products for the Conservation of Porous Building Materials in Monuments (In Science and Technology in Cultural Heritage), 19-21 June 1995,* 105-109. Rome, 1995.

_____. *Stone Conservation: An Overview of Current Research.* Santa Monica, CA: The J. Paul Getty Trust, 1996.

Price, Clifford A. "Stone - Decay and Preservation." In *The Association for Preservation Technology Masonry Conservation and Cleaning Handbook,* edited by Keith Blades, Gail Sussman, and Martin Weaver, 128-131. (location not given): The Association for Preservation Technology, 1984.

ProSoCo, Inc. "Product Data Sheet: H Consolidation Treatment." Kansas City, KS: ProSoCo, Inc., 1996.

_____. "Product Data Sheet: OH Consolidation Treatment." Kansas City, KS: ProSoCo, Inc., 1996.

_____. "Test Program for Stone Consolidation (Preservation)." *ProSoCo Technical Bulletin 483-2.* Kansas City, KS: ProSoCo, Inc., n.d.

Prueher, Brooks. "Consolidants, Coatings, and Water-Repellent Treatments for Historic Masonry: A Selected, Annotated Bibliography." *APT Bulletin* 26 (1995): 58-64.

Riecken, B., and B. Schwamborn. "Behavior of Impregnated Natural Stones after Different Weathering Procedures." *International Journal for Restoration of Buildings and Monuments* 2 (1996): 101-116.

Roby, Thomas C. "In Situ Assessment of Surface Consolidation and Protection Treatments of Marble Monuments in Rome of the 1980's, with Particular Reference to Two Treatments with Paraloid B72." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30*

234

September - 4 October 1996, edited by Josef Riederer, Volume 2: 1015-1028. Berlin: moller druck und verlag gmbh, 1996.

Rodrigues, J. Delgado, and A.E. Charola. "General Report on Water Repellents." *Science and Technology for Cultural Heritage* 5 (1996): 93-103.

Rodrigues, J. Delgado, and D. Costa. "Occurrence and Behavior of Interfaces in Consolidated Stones." In *Structural Studies of Historical Buildings IV - Volume 1,* edited by C.A. Brebbia and B. Leftheris, 245-252. Boston: Computational Mechanics Publications, 1995.

Rodrigues, Jose Delgado, and Elda De Castro. "Some Remarks on the Efficacy and Harmfulness of Stone Cleaning." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 1st International Symposium, Bari, 7-10 June 1989,* edited by F. Zezza, 491-494. Brescia, Italy: Grafo, 1990.

Rossi-Manaresi, R., A.E. Charola, A. Tucci, R.J. Koestler, and G.E. Wheeler. "Study of Accelerated Weathering of Limestones Treated with an Acrylic-Silicone Mixture." In *ICOM 7th Triennial Meeting,* 84.10.1-84.10.4. Copenhagen, 1984.

Rossi Manaresi, R., A. Rattazzi, and L. Toniolo. "Long Term Effectiveness of Treatments of Sandstone." In *Methods of Evaluating Products for the Conservation of Porous Building Materials, International Colloquium, Rome, 19-21 June 1995,* 225-244. Rome: ICCROM, 1995.

Roth, Dr. Michael. "Comparison of Silicone Resins, Siliconates, Silanes and Siloxanes as Water Repellent Treatments for Masonry." *ProSoCo Technial Bulletin 983-1.* Kansas City, KS: ProSoCo, Inc., nd.

Saleh, Saleh A., F.M. Helmi, M.M. Kamal, and A.-F. E. El-Banna. "Study and Consolidation of Sandstone: Temple of Karnak, Luxor, Egypt." *Studies in Conservation* 37 (1991): 93-104.

Sasse, H.R. "A New Chemical and Engineering Approach for Development and Optimization of Stone Protecting Materials." In *Science, Technology and European Cultural Heritage, Proceedings of the European Symposium, Bologna, Italy, 13-16 June 1989,* edited by N.S. Baer, C. Sabbioni, and A.I. Sors. London: Butterworth-Heinemann Ltd., 1991.

Sasse, H.R., and B. Schwamborn. "The Aachen Concept of Stone Preservation." In *EURO-MED Workshop on Contribution of Science and Technology to the Protection of Cultural Heritage in the Mediterranean Basin, 9-15 June 1997,* 46. Carthage, Tunisia, 1997.

Sasse, H.R., D. Honsinger, and M. Puterman. "The Aachen Concept: A New Technology in Stone Impregnation." In *1ˢᵗ International Colloquium Role of Chemistry in Archaeology, 15-18 November 1991,* 87-94. Hyderabad, India: Birla Institute of Sceintific Research, 1991.

Sasse, H.R., and R. Snethlage. "Methods for the Evaluation of Stone Conservation Treatments." In *Dahlem Workshop Reports: Saving Our Architectural Heritage, The Conservation of Historic Stone Structures, March 3-8, 1996,* edited by N.S. Baer and R. Snethlage, 223-243. Berlin: London: John Wiley & Sons, 1997.

Sasse, H. Rainer, and R. Snethlage. "Evaluation of Stone Consolidation Treatments." *Science and Technology for Cultural Heritage* 5 (1996): 85-92.

Schoonbrood, J.W.M. "Low Pressure Application Technique for Stone Preservatives." In *Conservation of Stone and Other Materials, Proceedings of the International RILEM/UNESCO Congress, Paris, 29 June-1 July 1993,* edited by M.-J. Thiel, 512-518. London: E & FN Spon, 1993.

Searls, Carolyn L., and David P. Wessel. "Guidelines for Consolidants." *APT Bulletin* 26 (1995): 41-44.

Selwitz, Charles. *Epoxy Resins in Stone Conservation.* Santa Monica, CA: The J. Paul Getty Trust, 1992.

_____. "The Use of Epoxy Resins for the Stabilization of Deteriorated Masonry." *APT Bulletin* 26 (1995): 27-34.

Sinner, P. "Conservation of Crystalline Marble with the Acrylic-Total-Impregnation Process." In *Science, Technology and European Cultural Heritage, Proceedings of the European Symposium, Bologna, Italy, 13-16 June 1989,* edited by N.S. Baer, C. Sabbioni, and A.I. Sors, 700-703. London: Butterworth-Heinemann Ltd., 1991.

Sleater, G. "Development of Performance Criteria for the Selection of Stone Preservatives." In *Decay and Preservation of Stone, Engineering Geology Case Histories Number 11,* edited by Erhard M. Winkler, 65-71. Boulder, CO: The Geological Society of America, 1978.

Sleater, Gerald A. "Stone Preservatives: Methods of Laboratory Testing and Preliminary Performance Criteria." *NBS Technical Note 941.* Washington, DC: U.S. Department of Commerce, National Bureau of Standards, May 1977.

Snethlage, Rolf, and Dietrich D. Klemm. "Scanning Electron Microscope Investigations on Impregnated Sandstones." In *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978.

Spry, Alan H. "Effectiveness of Preservatives and Surface Coatings in the Protection of Masonry." *ICCM Bulletin* 13: 65-72.

Stambolov, T. "Cleaning and Preservation of Stone Objects." In *The Treatment of Stone,* edited by R. Rossi-Manaresi and G. Torraca, 65-67. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1972.

Stambolov, T., and J.R.J. van Asperen de Boer. "The Deterioration and Conservation of Porous Building Materials in Monuments. A Literature Review." In *ICOM 7th Triennial Meeting, Copenhagen, 10-14 September 1984,* 84.10.23-84.10.24. Windsor, England: Reedpring Ltd., 1984.

Tabasso, M. Laurenzi. "Conservation Treatments of Stone." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 271-289. Paris: UNESCO, 1988.

_____. "Introduction to the Results of the Round Table Discussions." In *ICCROM International Colloquium: Methods of Evaluation Products for the Conservation of Porous Building Materials in Monuments (In Science and Technology in Cultural Heritage), 19-21 June 1995,* 51-56. Rome, 1995.

Tabasso, M. Laurenzi, A.M. Mecchi, and U. Santamaria. "Interaction Between Volcanic Tuff and Products Used for Consolidation and Waterproofing Treatment." In *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990,* edited by A. Elena Charola, 173-189. Rome: ICCROM, 1994.

236

Tabasso, M. Laurenzi, and U. Santamaria. "Consolidant and Protective Effects of Different Products on Lecce Limestone." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 697-707. Lausanne, Switzerland: Presse polytechniques romandes, 1985.

Tiano, P., C. Filareto, A. Granato, and F. Piacenti. "Methods and Materials Used for the Conservation of Monumental Works in Italy." In *8th International Congress on Deterioration and Conservation of Stone, Berlin, 30 September - 4 October 1996,* edited by Josef Riederer, Volume 2: 885-894. Berlin: moller druck und verlag gmbh, 1996.

Torraca, Giorgio. "General Philosophy of Stone Conservation." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 243-270. Paris: UNESCO, 1988.

_____. "Treatment of Stone in Monuments: A Review of Principles and Processes." In *The Conservation of Stone I, Proceedings of the International Symposium, Bologna, 19-21 June 1975,* 297-315. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1976.

Useche, L.A. "Studies for the Consolidation of the Façade of the Church of Santo Domingo, Popayan, Columbia." In *Lavas and Volcanic Tuffs, Proceedings of the International Meeting, Easter Island, Chile, 25-31 October 1990,* edited by A. Elena Charola, 165-171. Rome: ICCROM, 1994.

Van Gemert, D., D. Van Mechelen, and J. Dereymaeker. "Geo-Physical Testing of Ancient Masonry for Consolidation Purposes." In *Structural Conservation of Masonry, International Technical Conference, Athens 3 October - 2 Novemeber 1989,* 37-44. Rome: ICCROM, 1990.

Vassallo, J.C., and S.Z. Lewin. "Investigation of Polymer Impregnation of Stone I: In Situ Polymerization of Methyl Methacrylate." In *The Conservation of Stone II, The Preprints of the International Symposium, Bologna, 27-30 October 1981,* edited by Raffaella Rossi-Manaresi, 587-603. Bologna, Italy: Centro per la Conservazione delle Sculture all'Aperto, 1981.

Verges-Belmin, V., G. Orial, and R. Coignard. "Impregnation of Badly Decayed Carrara Marble by Consolidating Agents: Comparison of Seven Treatments." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 2nd International Symposium, Geneve, 19-21 November,* 421-437. Geneva: Museum of Art and History, 1992.

Weber, Dr. Helmut. "Conservation and Restoration of Natural Stone in Europe." *APT Bulletin* 17 (1985): 15-22.

_____. "Stone Conservation and Consolidation: Salzburg's Franziskanerkirche." *APT Bulletin* 19 (1987): 23-29.

_____. "Stone Conservation - Planning and Execution." *ProSoCo Technical Bulletin 483-1.* Kansas City, KS: ProSoCo, Inc., n.d.

Weber, Helmut. "Stone Renovation and Consolidation Using Silicones and Silicic Esters." In *The Conservation of Stone I, Proceedings of the International Symposium, Bologna, 19-21 June 1975,* 375-385. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1976.

Weber, Helmut, and Klaus Zinsmeister. *Conservation of Natural Stone: Guidelines to Consolidation, Restoration and Preservation.* Ehningen, Germany: expert verlag, 1991.

Weiss, Norman. "Chemical Treatments for Masonry: An American History." *APT Bulletin* 26 (1995): 9-16.

Wendler, E. "Materials and Approaches for the Conservation of Stone." In *Saving Our Architectural Heritage: The Conservation of Historic Stone Structures,* edited by N.S. Baer and R. Snethlage, 181-196. West Sussex, England: John Wiley & Sons Ltd, 1997.

Wendler, E., D.D. Klemm and R. Snethlage. "Consolidation and Hydrophobic Treatment of Natural Stone." In *Durability of Building Materials and Components, Proceedings of the Fifth International Conference held in Brighton, UK, 7-9 November 1990,* edited by J.M. Baker, P.J. Nixon, A.J. Majumdar and H. Davies, 203-212. London: E. & F.N. Spon, 1991.

Wheeler, G.S., S.A. Fleming, and S. Ebersole. "Evaluation of Some Current Treatments for Marble." In *The Conservation of Monuments in the Mediterranean Basin, Proceedings of the 2nd International Symposium, Geneve, 19-21 November,* edited by D. Decrouvez, J. Chamay and F. Zezza, 439-443. Geneva: Museum of Art and History, 1992.

_____. "Comparative Strengthening Effect of Several Consolidants on Wallace Sandstone and Indiana Limestone." In *7th International Congress on Deterioration and Conservation of Stone, Lisbon, Portugal, 15-18 June 1992,* edited by J. D. Rodrigues, F. Henriques, and F.T. Jeremias, 1033-1041. Portugal: Laboratorio Nacional de Engenharia Civil, 1992.

Wihr, Rolf. "Deep-Impregnation for Effective Stone-Protection." In *The Conservation of Stone I, Proceedings of the International Symposium, Bologna, 19-21 June 1975,* 317-318. Bologna: Centro per la Conservazione delle Sculture all'Aperto, 1976.

_____. "The Preservation of Damaged Stone by the Acrylic-Total-Impregnation Process: A Report on Seven Years Experimentation." In *The Deterioration and Conservation of Stone, Notes from the International Venetian Courses on Stone Restoration,* edited by Lorenzo Lazzarini and Richard Pieper, 303-312. Paris: UNESCO, 1988.

Zielecka, M., and P. Rosciszewski. "Water-Repellent and Structural Strengthening Agents for Masonry Materials." In *6th International Congress on Deterioration and Conservation of Stone, Torun, 12-14 September 1988,* edited by J. Ciabach, 641-644. Torun: Nicholas Copernicus University, 1988.

Zinsmeister, Klaus J.H, Norman R. Weiss, and Frances R. Gale. "Laboratory Evaluation of Consolidation Treatment of Massillon (Ohio) Sandstone." *APT Bulletin* 20 (1988): 35-39.

Structural Mechanics

Beer, Ferdinand P., and E. Russell Johnston, Jr. *Mechanics of Materials,* 2nd ed. New York: McGraw-Hill, Inc., 1992.

Berthelot, Jean-Marie. *Composite Materials: Mechanical Behavior and Structural Analysis.* New York: Springer-Verlag, Inc., 1990.

Dibenedetto, Anthony T. "The Suitability of Polymer Composites as Protective Materials." In *Conservation of Historic Stone Buildings and Monuments,* 312-321. Washington, DC: National Academy Press, 1982.

Hosek, J., and J. Panek. "Depth of Impregnation as the Criterium for Durability of Consolidated Stones." In *5th International Congress on Deterioration and Conservation of Stone, Lausanne, 25-27 September 1985,* edited by G. Felix, 801-807. Lausanne, Switzerland: Presse polytechniques romandes, 1985.

Jones, Robert M. *Mechanics of Composite Materials.* Philadelphia, PA: Taylor & Francis, Inc., 1999.

Venecanin, S.D. "Durability of Composite Materials as Influenced by Different Coefficients of Thermal Expansion of Components." In *Durability of Building Materials and Components, Proceedings of the First International Conference, Ottawa, Canada, 21-23 August 1978,* edited by Jane B. Wheeler, 179-192. Philadelphia, PA: American Society for Testing and Materials, 1980.

Walsh, Joseph B. "Deformation and Fracture of Rock." In *Conservation of Historic Stone Buildings and Monuments,* 87-107. Washington, DC: National Academy Press, 1982.

Concrete

American Concrete Institute. *Building Code Requirements for Structural Concrete and Commentary (ACI 318-95).* Farmington Hills, MI: American Concrete Institute, 1995.

Derucher, Kenneth N., George P. Korfiatis, and A. Samer Ezeldin. *Materials for Highway and Civil Engineers,* 3rd ed. Englewood Cliffs, NJ: Prentice Hall, 1994.

Popovics, Sandor. *Concrete Materials: Properties, Specifications and Testing,* 2nd ed. Park Ridge, NJ: Noyes Publications, 1992.

Testing

American Society for Testing and Materials. *Annual Book of ASTM Standards.* Volume 04.01, 1998.

  C 109/109M - 98: Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens).

_____. *Annual Book of ASTM Standards.* Volume 04.02, 1998.

  C 29 - 97: Standard Test Method for Bulk Density ("Unit Weight") and Voids in Aggregate.
  C 39 - 96: Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens.
  C 125: Terminology Relating to Concrete and Concrete Aggregates.
  C 127 - 88: Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate.
  C 128 - 97: Standard Test Method for Specific Gravity and Absorption of Fine Aggregates.
  C 136 – 84a: Standard Method for Sieve Analysis of Fine and Coarse Aggregates.
  C 192/C 192M - 95: Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.
  C 470 - 94: Standard Specification for Molds for Forming Concrete Test Cylinders Vertically.
  C 566 - 97 : Standard Test Method for Total Evaporable Moisture Content of Aggregate by Drying.
  C 617 - 94: Standard Practice for Capping Cylindrical Concrete Specimen.

Blackshaw, Susan M., and Susan E. Ward. "Simple Tests for Assessing Materials for Use in Conservation." In *Proceedings of the Symposium "Resins in Conservation" held at the University*

239

of Edinburgh, Edinburgh, United Kindom, 21-22 May 1982. Edinburgh: Scottish Society for Conservation and Restoration, 1983.

Clifton, James R. "Laboratory Evaluation of Stone Consolidants." In *Adhesives and Consolidants, Preprints of the Contributions to the Paris Congress, 2-8 September 1984*, 151-155. London: The International Institute for Conservation of Historic and Artistic Works, 1984.

Facaoaru, I., and C. Lugnani. "Contributions to the Diagnosis of Stone and Concrete Historical Structures Using Non-Destructive Techniques." In *Conservation of Stone and Other Materials, Proceedings of the International RILEM/UNESCO Congress, Paris, 29 June-1 July 1993*, edited by M.-J. Thiel. London: E & FN Spon, 1993.

Facaoaru, Ioan. "On the Use of Non-Destructive Methods in the Diagnosis and Assessment of Physico-Mechanical Residual Properties of Stone Masonry." In *Structural Conservation of Masonry, International Technical Conference, Athens 3 October - 2 Novemeber 1989*, 45-52. Rome: ICCROM, 1990.

Orton, Clive, Paul Tyers, and Alan Vince. *Pottery in Archaeology.* Cambridge: Cambridge University Press, 1993.

Reedy, Terry J., and Chandra L. Reedy. *Principles of Experimental Design for Art Conservation Research.* Santa Monica, CA: The J. Paul Getty Trust, 1992.

RILEM Group 25 P.E.M. *UNESCO-RILEM International Symposium on Deterioration and Protection of Stone Monuments.* Paris: Centre Experimental de Recherches et d'Etudes du Batiments et des Travaux Publics, 1978.

    Ultimate Compressive Strength, Test No. III.5.
    Water Drop Absorption, Test No. II.8a.
    Water Drop Absorption, Test No. II.8b.

Winkler, Erhard M. "Testing Techniques for the Effectiveness of Stone Consolidants." *APT Bulletin* 17 (1985): 35-37.

INDEX

243